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ENVIRONMENTAL TESTING OF CONTAMINANT PRODUCING MATERIALS FROM THE INTEGRATED LIFE SUPPORT SYSTEM

by W. S. Hodgkiss, Richard H. Johns, and James S. Swinehart

Prepared by
ATLANTIC RESEARCH CORPORATION
Alexandria, Va.
for Langley Research Center

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ABSTRACT

An experimental investigation has been made of the existing and potential airborne trace contamination composition within the Integrated Life Support System (ILSS) test chamber located at Langley Research Center. Selected materials from the ILSS were examined as probable sources of contamination under specific time and temperature conditions to determine their characteristic products of gaseous evolvement. Contaminated air samples were collected as both "whole air" samples and cryogenically trapped samples. Primary compound identifications were made by gas chromatography with verification by mass spectrometry and infrared spectrophotometry. Physical and chemical changes in the materials due to heat effects were examined by weight changes and differential thermal analyses.

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1.0 SUMMARY

Ten selected materials of construction of the Integrated Life Support System, Langley Research Center were exposed under specific time and temperature conditions in the presence of oxygen at 160 torr partial pressure. The purpose was to identify compounds given off by the construction materials which may be potentially toxic to occupants of the environmental system. The materials selected were plastic tubing, various types of insulations, and metal foil tape.

A test chamber was designed and used in the study as a means of providing a noncontaminant producing, stirred, nonflow atmosphere during the test series. Qualitative and quantitative analyses of the gas-off constituents are reported as carried out by gas chromatography with confirmation of identity where possible by mass and infrared spectrophotometry. Physical changes due to heat effects were measured by differential thermal analysis and by weight losses under specified conditions.

Cryogenically trapped samples of the atmosphere of the Life Support system were collected during preliminary test of the system and are reported as representative of the composition of the atmosphere of the chamber during that period.

2.0 INTRODUCTION

The components of the atmosphere of the closed system include a number of constituents which have been incorporated as a result of the outgassing of structural members and their coatings, heat degradation of lubricants, insulations, electrical components , etc. Since the concentrations of such components vary in respect to their toxic effects on humans, traces of one constituent may be more hazardous to an occupant of a closed system than a considerably larger concentration of another component.

An examination of the possible sources of such contaminants in the closed system recognizes that a single contaminant may be derived from a number of separate sources. Solvents from paints and coatings, plasticizers used to provide flexibility in tubing or insulations, lubricants used in moving parts are all potential sources of contaminants. In addition these materials may react with atmospheric oxygen. Heat effects, causing partial pyrolysis of some materials, can provide additional contaminants. Further, possibilities exist for oxidation of some of the gas-off constituents.

The Integrated Life Support System has, among other components, its own air purification system. This is designed to remove usual excesses of potentially toxic components from the atmosphere derived both from the materials of construction and from the occupants of the chamber. Since there is a finite time before contaminants can be removed by the air purification system, it is important to minimize or eliminate the contaminants which may not only contribute to physiological distress of the occupants but also to potential malfunction of apparatus or instruments in the chamber.

The study described here is concerned with the identification of specific constituents evolved into the gaseous environment from selected materials used in the construction of the Integrated Life Support System. Additional cryogenic trapping of constituents of the atmosphere of the system provide a preliminary measure of the identity and concentration of the components which may be considered the background of the system at its present stage of operation.

This report was prepared by the Atlantic Research Corporation under Task III of Master Agreement NASI-4425. The work was performed under the direction of the National Aeronautics and Space Administration, Langley Research Center, Langley Station, Hampton, Virginia and was administered by Charles H. Wilson, Applied Materials and Physics Section. The work reported here was carried out between January 25, 1966 and September 1, 1966.

Aiding the authors in the investigation were Robert Nugent, Ph.D., Mass Spectrometry; Susan Schwarz, Gas Chromatography; Roger Snyder, Laboratory Technician; and Leonard Goldstein, Laboratory Technician.

The work was carried out in the Analytical Chemistry Group, Advanced Technology Department of the Research Division.

3.0 DISCUSSION OF PROBLEM

The concentration of evolved contaminants is relatively small and avoidance of artifacts in a test program measuring small amounts of material is difficult and important. Several different materials of construction may evolve the same component. The amount given off by each material could be very small, but the sum from all sources could be above that necessary to cause physiological distress. Therefore, every possible step should be taken to detect as low a concentration of each compound as is possible even though this low level may be considerably below the concentration that causes physiological distress.

Since the concentration of some of the contaminants were very small extreme care was necessary to avoid the introduction of artifacts.

4.0 TYPES OF TESTS OF COMPONENTS IN THE ILSS

There were three distinct and separate series of tests to be performed under this task. Type A tests consisted of specimens subjected to the specified atmosphere at a temperature of 200°F for a period of 3 days followed by both qualitative and quantitative determinations of the resulting airborne contaminant species and the total weight changes of the original specimens. These tests were run with a static test vessel internal atmosphere. The Type B series of tests consisted of DTA-tests to determine the destruction temperature levels and were made under ambient environmental conditions. Analyses for decomposition products were not required in Series B tests. Third, or Type C, series of tests required the determination of gross weight changes in the original specimens produced by their subjection to their expected normal operating temperatures. The test atmospheres for the Type C series were identical with those for the Type A series with the exception that no stirring was required.

Series A tests will all be conducted at 200°F in the vessels and atmosphere specified in 2.1, Test Chamber. There will be a minimum of two replicates for each material. In the event that test results between the first two replicates differed by more than 20 per cent, a third replicate was made. Each specimen prior to testing was soaked in the specified atmosphere at 21°C for 24 hours and then weighed to within 0.1 milligram; the specimen was placed in the test vessel and the vessel flushed three times with the test air; the vessel was then charged with test air in such a manner that the subsequent adjustment of temperature to the required level resulted in the specified test atmosphere conditions; the temperature was adjusted to a temperature of 200°F and held for a period of 3 days; the test vessel atmosphere was sampled for contaminant analysis, after which the specimen was soaked in the test atmosphere for 24 hours and weighed to within 0.1 milligram. Each such cycle was considered a replicate requiring a separate test specimen.

Series B tests consisted of small, suitably prepared specimens

TABLE I

TEST SCHEDULE

Item No.	Material/Sample	Test Series-A				Test Series-B	Test Series-C			
		Specimen	Temp.	Press.	Time		Specimen	Temp.	Press.	Time
1.	Plastic tubing, formulation S-50 HL, clear	20 grams	200° F ±1° F	520 torr	72 hrs	Specimen size as required Pressure, air composition - ambient Time as required	20 grams	350° F ±3° F	520 torr	24 hrs
2.	Plastic tubing, Tygon B-44-4X	20 grams	200° F ±1° F	520 torr	72 hrs		20 grams	350° F ±3° F	520 torr	24 hrs
3.	Insulation, Min-K-1301	5 grams	200° F ±1° F	520 torr	72 hrs		5 grams	1200° F ±10° F		
4.	Insulation, 128 silicone coated glass cloth	30 grams					30 grams	350° F ±3° F		
5.	Insulation, silicone rubber and fiber glass white, cohrlastic 1015	30 grams					30 grams	350° F ±3° F		
6.	Tape, no. 425 aluminum foil, scotch brand	50 grams					50 grams	440° F ±4° F		
7.	Insulation, wire, Teflon MIL-W-16878, type E	10 grams					10 grams	150° F ±1° F		
8.	Insulation, JM-B10 Fiber	10 grams					10 grams	600° F ±6° F		
9.	Sponge insulation bonded with RTV	5 grams					5 grams	350° F ±3° F		
10.	Sleeve insulation	5 grams					5 grams	350° F ±3° F		

TABLE II

MATERIALS OF CONSTRUCTION EXAMINED

Item No.	General Description	Specific Description	Manufacturer or Supplier
1	Plastic tubing, Tygon	Formulation S-50HL, clear	U. S. Stoneware, Akron, Ohio
2	Plastic tubing, Tygon	" B-44-4X, clear	U. S. Stoneware
3	Catalytic burner and Bosch reactor insulation	Min-K-1301 [a]	Johns-Mansville
4	CO_2 concentrator and air evaporator insulation	128 Silicone coated glass-cloth, red, 6 lb/cu ft, Hamilton Standard Drawing No. 137X205-7H	H.I. Thompson Fiber Glass Co., Atlanta, Georgia
5	Waste dryer insulation	Silicone rubber and fiberglass, white, GD/C Spec.0-00589, cohrlastic 1015	Connecticut Hard Rubber
6	Adhesive tape	Micro Foil Sealing and Starting Tape No. 425 Aluminum Foil, Scotch Brand	3M Company, St. Paul, Minn.
7	Wire insulation	Teflon MIL-W-16878, Type E, GD/C [a] Spec. 0-69045	Common Stock
8	Insulation blanket	Unbonded "B" Fiber 4-1/2 Johns-Manville Certified B/O fiber	Thorpe Insulation Co. 4550 Federal Blvd. San Diego, California
9	Sponge insulation, CO_2 concentrator and air evaporator	AMS 3195 (Bonded with RTV 102)	AGC, Meridan, Conn.(or S. Halpert (8148 Ham.Std. Windsor Locks, Conn.
10	Sleeve insulation	Insulating sleeve used on [b] terminal lugs FCS B4-0779 GD/C part No. 87-85916	Aircraft Marine Products Inc. Harrisburg, Pa.
11(4b)	CO_2 concentrator and air evaporation insulation	Blue batting attached to Item No. 4	H.I. Thompson Fiber Glass Co., Atlanta, Georgia.

a. These samples were supplied completely by NASA Langley. Other samples obtained from supplier indicated in Column 3.

b. Sample obtained partially from supplier and partially from NASA Langley.

of each material run on a Aminco differential thermal analyzer. The amount of specimen will depend upon the type of equipment used. One replicate of each of the 10 materials was run under ambient environmental conditions, the DTA record taken up to the destruction temperature, and the resultant behavior analyzed. Destruction temperature is considered to be that temperature at which gross physical changes take place in the specimen, as, for example, melting, crumbling, burning, etc. No analysis of the degradation products was required. In addition some of the Series B tests were repeated on a Perkin Elmer differential scanning calorimeter.

Series C tests consisted of one replicate of each of the 10 materials. This series was designed to determine the gross weight loss of each material resulting from exposure to its normal operating temperature within the test atmosphere. Each test in this series was run for a period of 24 hours ±10 minutes. No determinations of air contamination was required for this series of tests. Each specimen prior to testing was soaked in the test atmosphere at 21°C for 24 hours and then weighed to within 0.1 mg, the test specimen was then placed in the test vessel containing the test atmosphere and the temperature adjusted to the approximate level and held for a period of 24 hours, the specimen was next soaked in the test atmosphere at 21°C for 24 hours and weighed to within 0.1 mg.

Following the above procedures there were30 tests in series A, 10 tests in Series B, and 10 in Series C.

5.0 TESTS OF ILSS ATMOSPHERE

Six (6) samples of air for analysis were collected from the ILSS at at two test conditions (three samples at each condition) over a period of two days. The samples were collected using the sample collection system developed for this purpose by the contract under Contract NASI-4425-1. An additional six samples were taken under similar conditions for use by LRC. Each sample set (three samples at a given test condition) consisted of a whole air"grab" sample, a sample trapped at -80°C and a sample trapped at -196°C.

Six (6) samples were analyzed qualitatively and used as a basis for quantitative analyses of the whole air samples. The data from the trapped samples were also be reported on a relative concentration basis for each component found.

6.0 TEST CHAMBERS AND PROCEDURE

6.1 DESIGN

To avoid introduction of artifacts the test chambers were fabricated entirely of stainless steel in such a way that no nonmetallic components come in contact with the test atmosphere. An assembled chamber is pictured in Figure 1. Each basic chamber is a two-liter 300 series stainless steel beaker to which a flanged head has been added. The head was fabricated from 0.5-in stainless steel plate and provided with a 2.5-in opening for access to the chamber. The opening is sealed by a bolted flange head, using a soft lead gasket.

The flange head, which comprises the lid of the test chamber, is provided with four openings which terminate in 0.25-inch Swagelock compression fittings. Two of the openings are fitted with Hoke Stainless Steel diaphragm valves for inlet and withdrawal of the test atmosphere. The remaining openings are fitted with a vacuum gage and a sheathed copper-constantan thermocouple, respectively. The disassembled apparatus is pictured in Figure 2.

The test sample is held in a 75-ml nickel crucible, which is supported in a ring suspended from the lid of the chamber. Stirring is accomplished by a magnetic stirring bar which is driven by a stirring motor beneath the test chamber.

Although the main use of the cylinders was in the Series A tests at 200°F, much higher temperatures could be used as required by the Series C tests. A few modifications however are required:

Above 220°F	Removal of pressure indicator and magnetic stirrers.
Above 610°F	Substitution of copper for lead in the gaskets.

6.2 PREPARATION

Prior to insertion of each specimen into the test vessel, the vessel was scrubbed with soapy water and rinsed thoroughly with distilled water. It was then rinsed twice each with methyl alcohol, acetone, and diethyl ether, successively. As an extra precaution the chambers were evacuated to less than 1 mm. after the ether rinse and before the nitrogen-oxygen purge. The test vessel was closed and purged with the test atmosphere at 200°F for a minimum of 4 hours after cleaning.

While at 200°F the test vessel was then evacuated again to 1 mm. The pressure was then increased to one atmosphere with the nitrogen-oxygen mixture. The evacuation and repressurizing were repeated twice at 200°F. The vessel was purged with the test atmosphere for an additional hour. The vessel was cooled to room temperature and the enclosed atmosphere checked by gas chromatography for residual solvents. If this check indicated more than 1 ppm for any single compound or more than 10 ppm for all residual compounds, the vessel was evacuated and heated until these limits were satisfied. In every cleaning procedure where the repeated evacuation and repressurizing were used, these limits for residual compounds were satisfied without further heating or evacuating.

Work on another program at Atlantic Research has shown that without the evacuation small but significant amounts of methanol that are adsorbed on stainless steel surfaces are not likely to be removed by the procedure given in the work statement. Testing after purging could show no methanol and significant quantities could still be adsorbed on the walls of the chamber. The methanol would be desorbed by gas-off products displacing it from the test vessel surfaces. This would cause a false identification of methanol as one of the gas-off products. Since the two methanol rinses serve no useful function in cleaning the chambers and may cause difficulty in the analysis, it is recommended that they be omitted in future work.

6.3 SAMPLE EXPOSURE

Series A tests were conducted at 200°F in an atmosphere of oxygen (160 ± 10 torr partial pressure) and nitrogen (360 ± 20 torr partial pressure). The gases were 99.6 mole per cent minimum purity and were oil-free and dry.

There was a minimum of three replicates for each material. Each specimen prior to testing was soaked in the test atmosphere at 21°C for 24 hours and then weighed to within 0.1 mg. The specimen was placed in the test vessel, and the vessel flushed three times with the test atmosphere. The vessel was then charged with test air so that the subsequent adjustment of temperature to 200°F resulted in the specified test atmosphere conditions. The temperature was held at 200°F for a period of three days (72 hours ± 5 min). The test vessel atmosphere was sampled for contaminant analysis, after which the specimen was then soaked in the test atmosphere for 24 hours and weighed to within 0.1 mg. Each such cycle was considered a replicate requiring a separate test program.

6.4 GAS SAMPLING

The following sampling technique was used to avoid contamination of the samples with any artifacts and to provide accurately measured volumes for more meaningful quantitative data. After exposure the chambers were cooled to room temperature. The pressure was increased to slightly above atmospheric with the nitrogen-oxygen test mixture; the increase in pressure was noted as accurately as possible. A Benton-Dickson all glass gas-tight syringe equipped with a #44305 stainless steel stopcock was used to withdraw 150-200 ml. The slight excess pressure in the exposure chamber expanded the syringe to 200 ml., and the valve was closed. A silicone rubber septum on a Luer-Lok fitting replaced the needle at the opening to the valve. The syringes were used to store the sample at atmospheric pressure. Since less than 10% of the atmosphere in the chambers was withdrawn and the pressure was slightly above the pressure during the exposure, the equilibrium was not significantly changed.

At no time was the pressure in the storage syringes ever less or greater than the ambient pressure. Further samples were drawn directly from the exposure chambers in 5-50cc Hamilton gas syringes; these later samples were used only to verify the qualitative analysis by establishing retention times. All quantitative analysis was done from the accurately measured volumes in the B&D storage syringes. The amounts withdrawn for these qualitative verifications were less than 3% of the total and the pressure was always slightly above the exposure pressure. Thus, the equilibrium was not disturbed significantly.

After all the samples were withdrawn for gas chromatography, the remaining contents of each exposure chamber were pumped into a liquid nitrogen trap. Mass spectra were obtained on each of these samples. The chambers were quickly evaucated to obtain the samples for the mass spectra and then quickly repressurized with the test atmosphere. The rapidity with which this was done precluded removing any significant materials from the test sample that were not already in the atmosphere. Attempts were made to trap various fractions from the gas chromatographs to obtain mass, infrared, and ultraviolet spectra. All these attempts failed because of the low concentrations of the compounds in the gaseous samples. The samples were equilibrated with test atmosphere for 24 hours and then weighed to the nearest 0.1 mg.

7.0 SERIES "A" TESTS

7.1 ANALYTICAL EQUIPMENT

The chromatographs that were used the most on this work were an F & M 810 chromatograph equipped with dual thermal conductivity and flame ionization detectors and a Perkin Elmer 800 dual flame ionization chromatograph. The Perkin Elmer and F & M chromatographs have proportional temperature control and can be temperature programmed.

The instrument and specific conditions used are indicated on each chromatogram in the tables summarizing the data and in the discussion. Since the information about the specific columns and programs is abbreviated in other parts of the report, a detailed list of those actually used is in Table III. Many other columns were tried and were not used for any of the analyses or gave no additional information.

All mass spectral data was obtained with a Bendix-Time-of-Flight Mass Spectrometer with a heated inlet system. Specific conditions are given with the individual spectra.

The infrared spectra were obtained by using a Perkin Elmer 186-0214 meter folded path gas cell and a Perkin Elmer 421 Spectrometer. More infrared spectra were not obtained because the ten meter gas cell did not arrive until the experimental work had almost terminated.

7.2 GENERAL PROCEDURES

The identification of the gas-off products was done by establishing retention times identical with those of known standards. This was the only way for positive identification as the contaminants were in too small a concentration for fraction collection. It is necessary in this approach to be certain that retention times be obtained on all compounds that are present. Compounds for retention time standards were selected according to the following criteria: (1) prior occurrence in space cabin atmospheres; (2) probability of being given off by materials studied; (3) mass spectral evidence from the spectrum of the test atmosphere after exposure. Since there is no possi-

TABLE III

COLUMNS AND PROGRAMS

Column No.	Liquid Phase Composition	%[a]	Support Composition	Size Length Diam.	Instrument	Program[b]	Carrier Gas[c] Gage Pressure psi	Carrier Gas[c] Flow rate ml/min
4	Apiezon	10	Chromosorb W	8' x 1'4"	F & M 810	70°I	40	40
5	C-400 Carbowax	20	Chromosorb W	12' x 1/4"	F & M 810	90°I	40	40
56a	UCON, HB-200	40	Chromosorb W	12' x 1/8"	PE 800	35°(1)→115°(25)	42	28
56b	UCON, HB-200	40	Chromosorb W	12' x 1/8"	PE 800	100°I	42	
58	1,2,3-tris (2-cyanoethoxy)-propane	4.5	Chromosorb P	6' x 1/4"	F & M 810	70°I	40	40
60	DEG	5	Chromosorb G Acid washed DMCS treated	24' x 1/8"	PE 800	55°C(5)→140(5.0)	46	32
61	QF-1	5	Chromosorb G Acid washed	12' x 1/8"	PE 800	65°(1)→135(2.5)	42	28
62			DMCS treated			55°(5)→145°(5)		
63	Silicone Grease	5	Haloport F	20' x 1/8"	PE 800	60°(4)→170°(3.75)	42	32
64	Triton X X-305	5	Chromosorb G Acid washed, DMCS treated	6' x 1/8"	PE 800	65°(1)→35°(2.5)	42	28

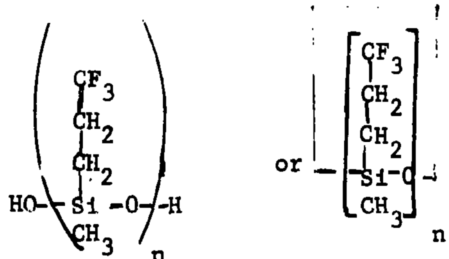
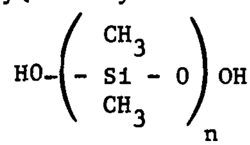
[a] Chemical names of liquid phases given in Table IIIb on following page.

[b] I = isothermal. On programed chromatographies the first temperature is the beginning temperature of the program and the number after it in parenthesis is the time in minutes the program was held isothermally at the starting temperature. The second temperature on the programed chromatographies is the temperature at which the increase in temperature was terminated and the number in parenthesis after the termination temperature is the rate of heating in degrees per minute. All temperatures for this table are in degrees Centigrade.

[c] Carrier gas is helium.

TABLE IIIb

COMPOSITION OF LIQUID PHASES

Name	Polarity[a]	Composition
Apiezon	Nonpolar (0.5)	Mainly long-chain branched and normal paraffins. A small amount of aromatic content
Carbowaxes and Ucon HB-200	Polarity (10)	$HO-(CH_2CH_2O)_nH$
DEG	Moderately polar (7.0)	Diethylene glycol succinate $H(OCH_2CH_2O_2CCH_2CH_2CO)_nOH$
QF-1	Slightly polar (3)	Poly(trifluoropropylmethylsiloxane) 
Silicone grease	Nonpolar (0.8)	Poly(dimethylsiloxane) 
Triton X-305	Moderately polar (5.5)	$C_8H_{17}C_6H_5O(CH_2CH_2O)H_{30}$

(a) Arbitrary polarity scale to indicate relative polarity from completely nonpolar (0) to very polar (high number).

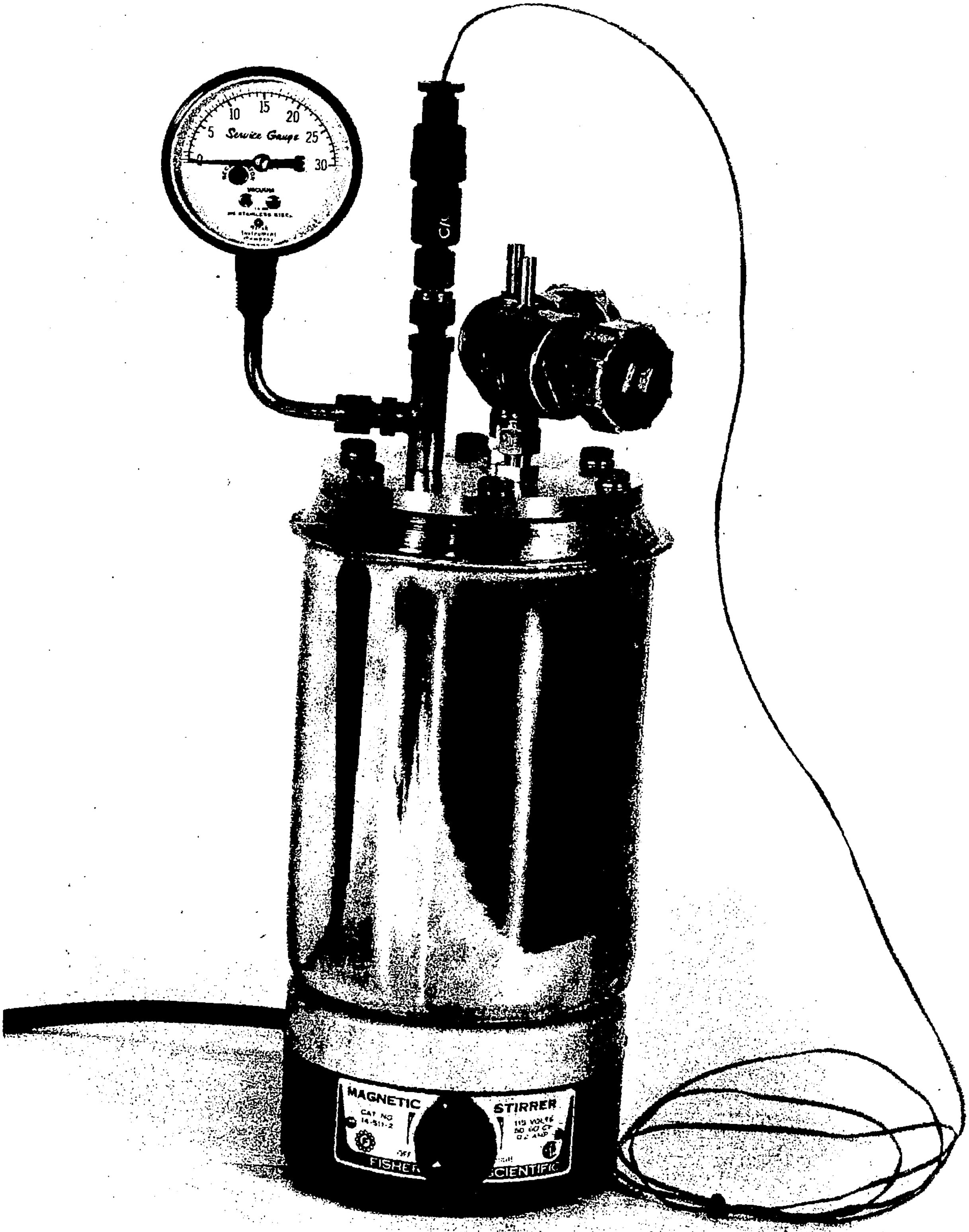


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Figure 1. Assembled Chamber.

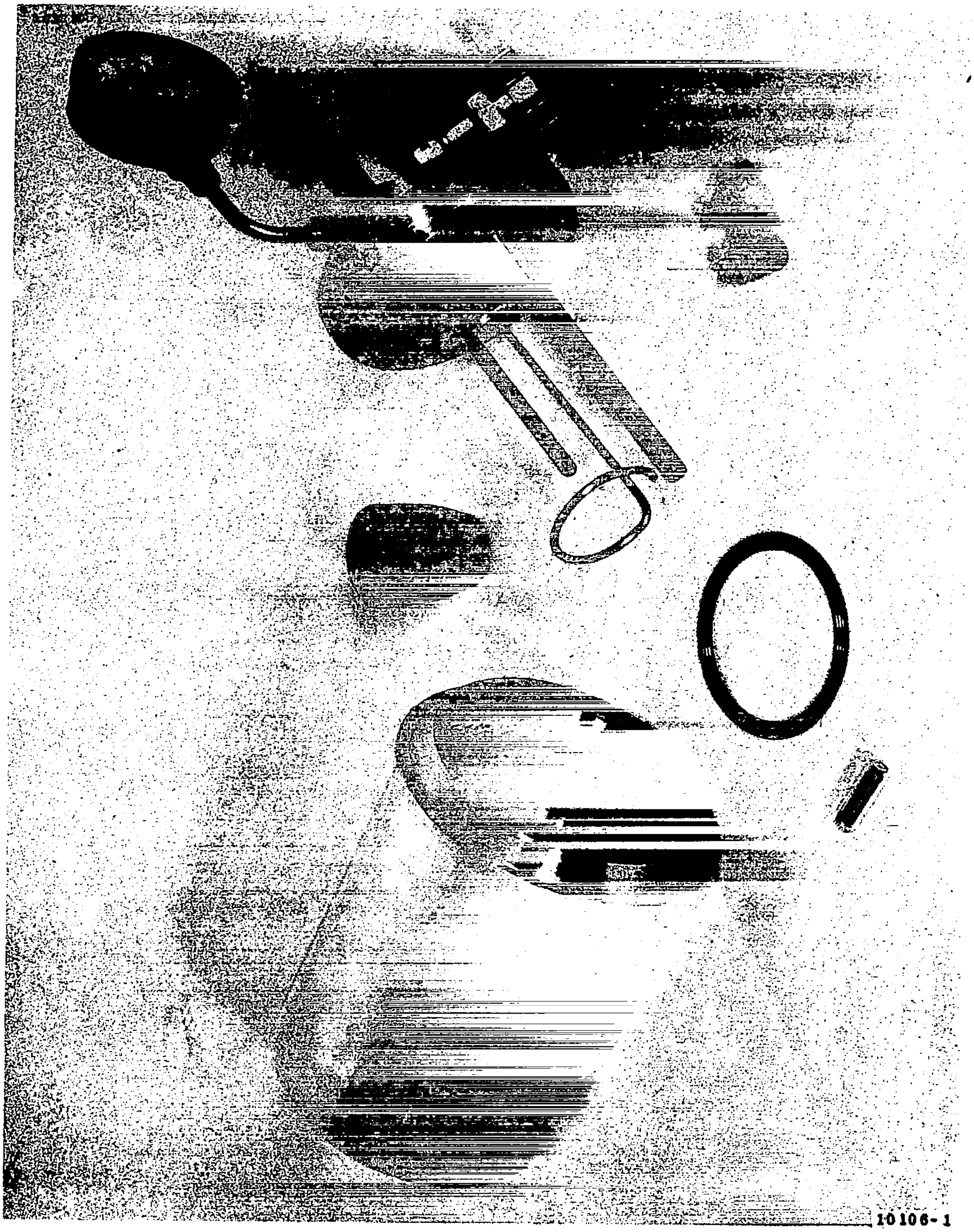


Figure 2. Disassembled Chamber.

bility of human expiration products, compounds that have been found in space capsules that were definitely and completely due to this source were not used as standards. Criterion 2 was not as useful as it could have been because the method of fabrication and composition of most the materials studied was proprietary and the firms involved were reluctant to disclose much information.

Most of the atmospheres after exposure contained a large number of compounds so that identity of most of these compounds by mass spectra alone was very difficult or impossible; however groups of peaks would indicate that certain compounds might be present and retention times were established for these compounds. The mass spectra were also useful in verification of compounds discovered initially by their retention times.

Standard retention times were also obtained on many compounds that were not indicated by the above three criteria. These were mainly compounds that were related (such as homologously) to the compounds selected by the above three criteria.

If retention times are used to identify compounds in complex mixtures, the retention times of the compounds on a given column with a certain program must not vary more than 5% and preferably less. If the columns are operated below overload conditions, retention times will vary only because of 1) change of column, 2) variation of temperature, 3) variation of flow.

The principal changes of a gas chromatographic column are caused by:

A) Bleeding of liquid phase

B) Decomposition or other chemical changes of the liquid phase

C) Deposition of relatively nonvolatile materials in the liquid phase.

A and B were minimized by aging the columns at 25°C below maximum recommended operating conditions for at least 65 hrs with an inert gas passing through at a flow rate of at least 15 ml/min. The columns were "steam cleaned" by three 0.2 ml injections of water after 4 hrs, 63 hrs., and 64 hrs. of aging. For isothermal operation, the columns were then always operated at least $40^{\circ}C$ below the aging temperature. For programed operation, the upper limit of the temperature program was never higher than $20^{\circ}C$ below the aging temperature. For example, the QF-1 (maximum recommended temperature $226^{\circ}C$) column was aged at $200^{\circ}C$. The column was used on two programs. The highest temperature limit was 145°; however, after this temperature limit was reached, the column was operated isothermally so that $40^{\circ}C$ below aging limit was applied. Many of the columns were lightly loaded with liquid phase. Light loading decreases the change in retention time due to bleeding. Columns that were heavily loaded were operated even further below their aging temperature. For example, a 40% Ucon (maximum temperature 200°) column (which was probably much less loaded than that after aging) was aged to $175^{\circ}C$. It was operated isothermally at $95^{\circ}C$ and programmed to a maximum temperature of $115^{\circ}C$.

Changing of the columns due to deposition of high boiling material was eliminated or at least greatly minimized by using redistilled standards free of such nonvolatile compounds, by injecting small samples of the standards and by "steam cleaning" the columns with 10 μl of water after retention times on a large number of standards had been determined. The use of small sample size and very pure standards also decreased the possibility of chemical reaction between the stationary phase and the sample.

Often the problem of drifting absolute retention times caused by small temperature and pressure variations is ignored, and retention times relative to some internal standard are used. The original intention in this investigation was to use relative retention times, and some of the first standards were reported in both relative and absolute retention times (Letter Report #2, p.4, April 12, 1966). The injection of internal standards into the gaseous unknowns in amounts small enough not to mask some of the gas-off products and large enough to register proved difficult. This caused the abandonment of the use of relative retention times.

The drift of absolute retention times due to temperature and flow variations was eliminated by: (1) Using the best equipment available; this included proportionally temperature controlled ovens and precision flow control and metering devices, (2) Allowing twenty minutes after cooling on the programed chromatograms so that the column packing, column and not just the oven air, could equilibrate to the desired operating temperature, and (3) the use of "external" standards.

A standard whose retention time had been determined on the first day of use of the column was rechromatographed at the beginning and the middle of each day the column was used. Most of the time the retention times of these "external" standards agreed within 2.5% of the retention times determined on the first day of use. On the days the retention time varied by more than this, the temperature of the oven was verified by use of a Leeds and Northrup potentiometer with an iron-constantan thermocouple. If the temperature of an isothermal chromatogram or the beginning of a programmed chromatogram varied by more than 0.3^{o} from the temperature of the oven on the first day, the oven temperature was adjusted to the original value $\pm 0.1^{o}C$. If the retention time of the external standard varied and the oven temperature was within the limits stated above, the flow rate was adjusted for coincidence of retention times. It was found that: (1) The variations in retention times of the external standard were small. (2) When adjustment was required, it was usually the flow that needed changing. (3) There was no more tendency for the retention times to drift at the end of the investigation than at the beginning indicating that the columns had not changed significantly.

The mixtures were analyzed quantitatively by comparing peak heights of the samples with peak heights obtained from known quantities of the same compounds on one of the columns used for qualitative analysis. At first the quantitative standards were gas samples diluted with the nitrogen-oxygen test atmosphere, (Letter Reports 2,3, and 4). It was found that calibrations obtained this way were not as precise as those obtained from dilute liquid solutions. The higher precision from use of dilute liquid was probably due to the very small sample sizes that could be injected with liquids (but not with gases) and adsorption on the surface of the containers used for dilution of the gaseous sample.

Tables IV - XIII, indicate the compounds identified and the method used to identify them. A compound was not considered identified until its retention times were found to be the same as the retention times of a known standard on at least two different stationary phases,or very strong mass or infrared spectral evidence for the compound and identical retention times on at least one liquid phase were obtained. In addition, if a compound is indicated as definitely or probably being present, <u>none</u> of the mass spectral or gas chromatographic data could indicate that it was not present. For example, if there was a peak with the same retention time as a known standard on as many as five different stationary phases, and the unknown had no peak on a sixth stationary phase where a compound of its concentration should have a peak and there was no possibility of masking by other strong peaks or by the background, then it was concluded that the unknown was not the compound in question. Similarly if <u>all</u> the gas chromatographic data indicated a relatively <u>large</u> amount of a given compound but the principal peaks of this compound could not be found in the mass spectrum of the sample, the compound was considered not present. When this occurred, a new search was begun for a compound consistent with both the mass spectral and the gas chromatographic data. In many cases no more confirmations of the unknown could be obtained on the liquid phases attempted because of low concentration and/or because of masking by components of higher concentration or by high background. For such non-confirmations NC is written in Tables IV -XIII This is not to be considered a negative result. As indicated above, if a negative result was found for a proposed compound, the compound was not entered in the tables and a new search was begun for the unknown responsible for that peak.

A compound is recorded as definitely being present (D) if the minimum requirements (outline above and in the work statement) were met plus at least one more retention time coincident with that of the known standard. If only the minimum requirements were met the compound was recorded as probably being present (P). If less than the minimum requirements were met but evidence indicated that the compound might be present the contaminant was recorded as possibly being present (M). If it seemed very unlikely that the compound would be given off by the substance being tested the requirements for P, M, and R were increased by one additional retention time.

For most of the compounds identified there were at least three retention times identical with a known standard and spectral verification or four retention times identical with a known standard.

For the ten exposure experiments the duplicate samples showed good agreement qualitatively but differed sufficiently quantitatively that it was necessary to do triplicate exposures for each sample. One sample, solder lug insulation, was done in quadruplicate because of the need for extra material to obtain a mass spectra.

In general the triplicate samples agree fairly closely. The principal variation was the high concentration of carbon tetrachloride found from some of the first exposures and not from the others. A possible cause of this is from the dessicator in which the samples were soaked. The dessicator grease had been removed with the aid of carbon tetrachloride. Even though a thorough attempt was made to remove all traces of carbon tetrachloride, apparently a sufficient amount remained to contaminate the first set of samples but not the second or third.

7.3 MASS SPECTRAL TECHNIQUES

Samples for the mass spectra analysis are taken from the exposure chambers following the gas chromatographic sampling. The sample is introduced into the mass spectrometer through a liquid nitrogen cooled loop. This allows for selective trapping to exclude most of the atmosphere in the test sample by not trapping the components that are not condensed by the liquid nitrogen cooled loop. After selective pumping, the trap is warmed by a heat gun to introduce a sample of approximately $P_{TC} = 110\mu$ for the test run. The spectrum is then copied and peak heights for the occurring m/e peaks are measured. The background read on the day of the test run under the same instrumental conditions is deducted. The trapped carbon dioxide, which is a large component in most samples, is deducted on the basis of calibration data for contribution at specified m/e numbers. The basis for the quantity peak height attributed to carbon dioxide at its maximum peak of m/e = 44 is explained in footnotes at the end of each mass spectra data table. The trapped water and the contribution from the oxygen, nitrogen and argon (if present) is deducted in the same manner. The remaining deflections at specified m/e numbers are listed in the data tables and reduced to percentage of the primary remaining peak. This data is listed in Tables VI - IX.

7.4 RESULTS, GAS CHROMATOGRAPHY AND MASS SPECTRA

In Tables IV - XIII the first column is the name of the contaminant, the second column refers to degree of identification, the third column is the concentration of contaminant, the remainder of the columns except the last one show the retention times used to identify the unknowns. The last column gives the principal peaks used for mass spectral verification. The abbreviations used are explained above and in footnotes to the tables. Infrared spectra are discussed after the Tables (Section 7.4).

TABLE IV

IDENTIFICATION OF COMPOUNDS FROM TYGON, #1

Compound	Certainty of identification	Average mg. 10^{-6} per g. of material	Retention times: Carbowax 5	Apiezon 4	1,2,3-Tris-cyano 58	QF-1 61	DEGA 60	UCON 56a	UCON 56b	Silicone grease 63	Mass spec. verification
Acetaldehyde	P	7266	3.65	1.20	NC	NC	NC	NC	NC		44, 43, 42, 29, (-30)
Acetone	D	< 25000	5.25	1.8	13.0	2.45			3.4	2.0	58, 57, 43, 29, 15
Benzene	P	< 3500	7.3	NC	13.40			7.05?			78, 77, 76, 75, 74, 52 51, 50
Butane	M		2.1	1.4	0.68						
1-Butanol	M		23.1	3.9		NC	18.47				
Butyraldehyde	M		6.6	2.8							72, 43, 42, 29
Carbon tetrachloride	D	3430	5.25	4.7	5.6	2.40			5.5		121, 119, 117, 86, 85, 84, 83, 82, 72, 70, 44, 47, 38, 37, 36, 35
Cyclohexane	P	2959	5.1			2.3?		3.47		6.95	
Diethyl ether	P	866	2.8	1.9	2.10					2.01	
Ethanol	D	2673	9.25		15.2	1.36	9.62	7.05	NC	2.0	32, 31, 29, 28
Ethyl acetate	M		6.5					5.90			
Ethyl benzene	M		17.6			NC	12.6			16.19	
Heptane	P	469	2.9	5.5	1.60			3.47		8.43	
Hexane	P	589		NC	1.18	1.47		1.90			
1-Hexene	M		2.8	2.8	1.4						84, 83, 32, 41,
2-Hexene	P		2.8	3.3	1.4	1.51	4.0				84, 83, 55, 29, 15
Methanol	P		7.35	1.20	13.2	NC	7.79	4.30	12.5	1.23	32, 31, 29, 28
Methylcyclohexane	P	1417	3.65	NC	2.6	2.45		NC		9.7	
Methylcyclopentane	P	90	2.9	3.9	1.9					3.80	
Methyl acetate	P	< 530		1.9	10.05		7.79	4.30			
Methylene chloride	P	40	6.6	1.8?							
Methyl ethyl ketone	D	1354	7.3	2.7	19.2	4.07	12.58	3.47	5.9	3.84	72, 57, 43, 29, 15
Octane	P	483	NC	11.7	2.2		6.10			13.59	
2-Octene	M		4.7	11.7	3.25, 3.75		9.6		7.0		
Pentane	P	1316		1.85	0.9	1.13					
2-Propanol	P	428	NC	1.9	13.3	1.36	9.6				
Toluene	P	1.5	12.07			4.25	10.93				

TABLE V

IDENTIFICATION OF COMPOUNDS FROM TYGON, #2

Compound	Certainty of identi-fication	Average mg. 10^{-6} per g. of material	Retention Times: Carbowax 5	Apiezon 4	Triscyano 58	QF-1 62	UCON 56a	Triton 64	UCON 56b	Mass Spec. Verification
Acetaldehyde	P		3.7	1.2	NR	NR	NR	0.72	1.10	44, 43, 42, 92, (-30)
Acetone	D	~500	5.22	1.80	13.00	2.69	3.57		3.4	58, 57, 43, 29, 15
Benzene	M		7.31		13.00	2.62				
Butane	M		2.1	1.5	0.64					
1-Butanol	P			3.97		3.22	18.67	4.67		
Butyraldehyde	M		6.6	2.6	NR	NR	NR	NC	NC	72, 43, 42, 29
Carbon tetrachloride	D	3860	5.25	4.7	5.70		9.45		5.1	121, 119, 117, 86, 85, 84, 83, 82, 72, 70, 49, 47, 38, 37, 36, 35
Cyclohexane	P	< 6		5.09	2.32	2.11	3.57			
Diethyl ether	P	356	2.86	1.87	2.1					
Ethanol	D	~550	8.96	1.87	14.70	1.52	6.80		NC	
Ethyl acetate	M			2.61		3.22	6.04			
Ethyl benzene	M		17.39				18.67	5.40		
Hexane	P	312	NC	NC	1.15	1.52		0.50		
1-Hexene	P		2.85	2.6	1.25					84, 83, 57, 56, 43, 41
2-Hexene	P		2.85	2.6	1.25				NC	84, 83, 57, 56, 55, 29, 15
Methanol	P		7.3	1.16	12.9	1.2		0.74	NC	32, 30, 29, 28, 15
Methyl acetate	M	<31	5.25	1.87	NC	2.11				
Methyl ethyl ketone	D	586	7.31	2.65	19.00	3.83	6.07		5.9	72, 71, 57, 43, 42
Methylene chloride	P	630	6.50	2.02	9.32		5.41			
Methyl formate	M		3.92			1.52		0.74		
n-Octane	D	175	3.9	12.69	2.1	3.22	6.07			
2-Octene	D		5.25	3.3					7.0	57, 56, 55, 29, 15
Pentane	P	1423			0.95	1.30		0.50		
2-Propanol	D	~ 800	9.12	1.87	13.00	1.52	6.80			
Toluene	P	292	12.2	9.79				3.3		
n-Heptane	D	254		5.40	1.61	1.99	3.57	0.74		
Methylcyclohexane	P	611	[illegible].92		2.60	2.70	5.41			

TABLE VI

IDENTIFICATION OF COMPOUNDS FROM MIN-K FIBER, #3

Compound	Certainty of identi-fication	Average mg. 10^{-6} per g of material	Retention times: Carbowax 5	Apiezon 4	Triscyano 56	QF-1 62	DEGA 60	UCON 56a	Silicone Grease 63	Triton 64	Mass Spec. Verification
Acetaldehyde	M	< 110	3.8	1.2							43, 42, 29
Acetone	D	~ 800	5.29	2.05	13.20	2.58		3.68	2.29		58, 57, 43
Acrolein	M		5.29	1.45					1.61		
Benzene	D	275	7.74	4.70	13.20	2.58		7.36		1.72	78, 52, 51, 50
Butane	P	1756	2.05	1.45	NC				1.61		
Diethyl ether	M	< 10	2.7	NC	2.00				2.29		
Ethanol	P	268	9.37	NC	14.30				2.09		
Formaldehyde	M	< 220	3.68?	1.4							30, 29
Hexamethylcyclotrisil-oxane	M			NC	5.32		11.08	7.36			
Hexane	M	< 10	2.7	3.06							86, 57, 56, 45, 42, 41
Methanol	P	< 329	7.74	1.18	12.50				1.61		
Methyl acetate	P	< 110	5.29	2.05	NC			3.68			
Methylene chloride	P	< 110	6.5	2.05	8.85				2.29		86, 84, 83, 51, 49, 47, 37, 36, 35
n-Pentane	P	~700	2.12	2.05	0.94		1.11				72, 71, 57, 56, 43, 42, 41.
Toluene	P	329	12.10	9.4			11.08		10.65	3.20	92, 91
Trichloroethylene	M		8.69						7.48	2.37	

TABLE VII

IDENTIFICATION OF COMPOUNDS FROM SILICONE GLASS CLOTH,#4

Compound	Certainty of identification	Average mg. 10^{-6} per g of Sample	Retention times: Carbowax 5	Apiezon 4	Triscyano 58	QF-1 62	QF-1 61	Silicone Grease 63	DEGA 60	Triton 64	Mass spec. verification
Acetone	D	< 350	5.36	1.97	13.30	2.40	2.52	2.24			58, 43
Acrolein	M		5.36					2.32?	8.10		
Benzene	D	< 250	7.75	4.58	13.30	2.40	2.60	5.87	13.38	1.77	79, 78, 77, 76, 74, 52, 51, 50, 39, 38, 37
Carbon tetrachloride	P	< 275	5.36	4.58	5.4			6.08			
Ethyl acetate	P		6.58			3.15		4.17	9.92		
Dimethyldimethoxy-silane	P		3.93	3.13	2.96	2.40					
Diethyl ether	P	< 6	2.6	1.97	2.08			2.24			
Ethanol	D	121	9.11	NC	14.7		NC	2.28?	9.92		46, 45, 31, 30, 29, 27
Ethyl formate	P	< 20		NC	9.0	1.86	2.01	2.91			74, 31, 29, 27, 26
Hexane	P	< 2	2.60	3.13	1.14	1.47					43, 42, 41, 5
2-Hexene	M		2.60	3.13							84, 69, 55, 42, 41
Methanol	P	< 9	7.75	NC			1.23		8.10	1.07	31, 30
Methyl acetate	P					2.40?	2.01	2.85	8.10		
Octane	P	< 8	3.93			3.15		13.95			
Pentane	D	< 618			0.93	.98	1.17	2.85	2.79	0.58	
Propionaldehyde	P			1.97				2.24		0.92	
Propanol	M						2.01	2.85		2.74	

TABLE VIII

IDENTIFICATION OF COMPOUNDS FROM COHRLASTIC, #5

COMPOUND	Certainty of identification	Average mg. 10^6 compound per g. of Material	Retention times Carbowax 5	Apiezon 4	1,2,3-Tris-Cyanopropane 58	QF-1-OLD 61	UCON 56b	Times UCON 56a	TRITON 64	Mass spec. verification
Acetaldehyde	P	~300	3.8	1.3					0.80	44, 43, 42, 29, no 30
Acetone	D	~400	5.26	1.58	12.86	2.63	3.5		1.03	58, 57, 43, 29, 15
Benzene	P	< 10	7.6	4.6	14.40	2.62			1.75	78, 77, 76, 75, 74, 52, 51, 50
Butane	P	< 11	2.1	1.58	0.75					
Carbon tetrachloride	P	~300	5.25	4.7	5.4					121, 119, 117, 86, 85, 84, 83, 82, 79, 72, 70, 49, 47, 38, 37, 36, 35.
trans-1,2-Dimethylcyclohexane	M		4.9	15.0						
Ethanol	D	48170	9.1	1.70	14.4			6.73	1.4	46, 45, 43, 29, 31
Ethyl formate	P	< 80	1.85		9.0		3.55			74, 73, 56, 55, 45, 29
Formaldehyde	P	< 400	3.7	1.4						
Freon TF	M			2.0					0.42	
Hexamethylcyclotrisiloxane	P		NC	7.2	5.5			5.77		
2-Hexene	M		2.70	NC	1.7					84, 83, 55, 55, 29, 15
Methanol	D	< 10	7.82	1.3	12.5			4.93	1.07	
Methylcyclohexane	P	30	3.8	7.6		2.50				
Methylene chloride	P	< 140	6.4	2.0	9.1					86, 85, 84, 83, 82, 80, 51, 49, 48, 47
Methyl ethyl ketone	P	< 22	7.2	2.7		4.07			1.43	72, 71, 57, 43, 29, 15
Pentane	M	< 11		1.98		1.15				

TABLE IX

IDENTIFICATION OF COMPOUNDS FROM ALUMINUM FOIL, TAPE #6

COMPOUND	Certainty of identification	Average mg. 10^{-6} per g of material	Retention times: Carbowax 5	Apiezon 4	Triscyano 58	QF-1 61	UCON 56a	Mass spec. verification
Acetone	D	250	5.27	2.00	12.90			58, 57, 43
Benzene	P		7.31	4.66	NC		7.67	78, 76, 52, 51, 50, 39, 38, 37
Butane	M	95	2.05	Possible	.53	Possible		58, 57
Cyclopentane	P	39	3.32	2.91			2.23	69
Carbon tetrachloride	D	724	5.27	4.66	5.35		9.37	
1,2-Dichloroethane	M		13.86?	3.86		2.09		
Dimethyldimethoxysilane	M		3.81	2.91			4.69	
Ethyl acetate	P		6.46		12.90	2.99		88, 87, 73, 57, 31, 15
2-Ethyl-1-hexene	D		3.81	8.23	3.3	2.73	4.79	97, 69
n-Heptane	D	< 30	3.10	5.5	1.47			
2-Heptene	D		3.32	5.6	2.51		4.30	98, 97, 55, 43,
n-Hexane	D	29	2.76	2.91	1.13	NC	2.23	57, 41, 42, 43
1-Hexene	D	< 34	3.10	2.9	1.47	NC	NC	84, 83, 56, 41, 42, 43
Isopropyl alcohol	D	614	8.66	2.00	12.90			45, 43, 41, 42, 29, 27
Methyl acetate	P	< 15	5.27	2.00			4.30	74, 73, 59, 43, 31, 15
Methylcyclohexane	D	< 79	3.81	7.37	2.51		4.79	83
Methylcyclopentane	D	28	2.76	3.86	1.88	1.83		83, 69
Methyl ethyl ketone	D	21	7.31	2.91	18.75			72, 71, 57, 43
2-Pentene	M		2.41		1.47		1.59	70, 69, 57, 43
2-Octene	M				3.70	2.99	7.67	57, 55
Toluene	P	< 77	11.68	NC	NC		2.1	91

TABLE X

IDENTIFICATION OF COMPOUNDS FROM TEFLON WIRE INSULATION , #7

Compound	Certainty of identification	Average mg. 10^{-6} per g of material	Retention times Carbowax 5	Apiezon 4	Triscyano 58	CF-1 61	CF-2 62	DEGA 60	UCON 56b	Silicone grease 63	Mass spec. Verification
Acetaldehyde	P	<58	3.68	NC	NC		NC		NC	1.50	29, 44
Acetone	D	~660	5.12	2.02	12.8		3.19	8.99	3.73	2.06	43, 58
Benzene	P	< 12	7.2	4.72	NC		NC	8.99	NC	NC	78, 79, 77, 76, 52, 51, 50
Chloroform	P	583	NC	3.17	12.8		NC		NC	4.28	
Dimethylcyclohexanes	M		5.12	NC	NC					NC	112,97, 85, 84, 83, 70, 69, 68, 57, 56, 55, 54, 44, 43, 42, 41, 40, 39
Ethanol	M	< 150	8.9	NC	NC		NC		NC	2.06	31, 32, 30, 29, 28
Ethyl acetate	P		NC	2.72	12.6	3.08	3.19			4.28	
n-Hexane	P	< 6	2.55	3.17	NC		NC		NC	NC	57, 56, 43, 42, 41, 39, 29, 27.
Methanol	D	~1500	8.9	2.02	12.8		NC		NC	1.50	32, 31
Methyl acetate	P	< 40	5.12	2.02	NC		2.15		4.09	NC	
Methylcyclohexane	P		3.89	7.01	2.84			8.99		NC	97, 83
2-Octene	P		5.10	NC	3.42	3.08	NC	8.99	8.43	NC	84, 69, 56, 55, 43, 42, 41, 29, 27
Pentane	M	<161	2.32	2.02	NC		NC	2.51	1.00	NC	
2-Propanol	P	<350	8.9	2.02	12.8		NC		NC	NC	45, 43, 41, 42, 29, 27

TABLE XI

IDENTIFICATION OF COMPOUNDS FROM UNBONDED B-FIBER , #8

COMPOUND	Certainty of identification	Average mg. 10^{-6} per g of material	Retention times: Carbowax 5	Apiezon 4	Triscyano 58	QF1 61	UCON 56a	Silicone 63	Triton 64	Mass spectra verification
Acetone	D	<632	5.29	1.85	13.12	2.52	3.8	2.09	1.05	58, 57, 43
Benzene	P	<402	7.40		13.12	6.40				
1-Butanol	M			3.81		3.23			4.86	
Dimethyldimethoxysilane	P		3.84	3.35	2.92					
Ethanol	P	119	9.15	1.85				2.09	5.38	46, 45, 31
Ether	P	< 97	2.70	1.85	2.06			2.09		
Ethyl acetate	P		6.50	2.83	13.12	3.23				
n-Heptane	P	134		5.7	1.4		3.3			
2-Heptene	P		3.84	5.7	2.06					55, 53
n-Hexane	P	<27.3	2.70	3.35	1.08					
Methanol	D	25.5	Possible	1.42		1.15		1.49	1.05	31
Methyl acetate	D	948	5.29	1.85	9.42			2.74	1.05	74, 74, 59, 43, 31, 15
Methylcyclopentane	P	< 5.6	2.70	3.80	2.06					
Methyl ethyl ketone	D	88	6.50	2.83		4.10				72, 71, 57, 43
Methyltrimethoxysilane	M		7.40	4.18				1.60		
n-Octane	P		3.84			3.23		13.51	1.05	
n-Pentane	P	< 5.36			1.08	1.07		2.74		
Propionaldehyde	P		5.29	2.83				2.09		
Tetrachloroethylene	P		9.15			4.38		13.51	3.5	

TABLE XII

IDENTIFICATION OF COMPOUNDS FROM SPONGE INSULATION BONDED WITH RTV, #9

Compound	Certainty of Identification	Average mg.10^6 per g. of Material	Retention Times: Carbowax 5	Apiezon 4	Triscyano 58	QF-1 6	DEGA 60	Silicone Grease 63	Ucon 56a	Triton 64	Mass Spec. Verification
Acetone	D	< 54	5.34	1.87	5.34	2.87	8.41		2.82		
Acrolein	M		5.4	1.87			8.41				
Benzene	D	< 22	7.65	4.6	13.6			5.69	8.05		79, 78, 77, 76, 52, 51, 50
n-Butane	M		2.14	1.47				1.53			
Butyraldehyde	M		6.57	2.75							
Cyclopentane	M	< 10	2.77	2.7							
Ethanol	P	357	9.1	NC	14.7(?)						46, 45, 31, 30, 29, 27
Ethyl Acetate	M		6.57	2.6	12.1						
2-Heptene	M			5.47	2.23				3.82		
Hexamethylcyclotrisiloxane	M			7.4				13.67	6.51		
n-Hexane	M	< 8	2.77	3.16	1.1						
Methanol	D	< 54	8.86	1.12	12.0	1.16		1.53		1.08	31
Methyl ethyl ketone	P	< 108		5.7	14.2				6.51		
Methyl formate	M		3.84			.93		1.53			
n-Octane	D	< 76	3.84		2.23	2.78		13.67	6.51	1.10	
2-Octene	P		4.75		3.10		8.41		8.05		
n-Pentane	P	209	2.19	1.87		1.16	2.72				
2-Propanol	M	< 618	8.99	1.87							
2-Propyl acetate	M			3.16	11.94			5.69	6.51		

TABLE XIII

IDENTIFICATION OF COMPOUNDS FROM SOLDER LUG INSULATION, #10

Compound	Certainty of identification	Average mg. 10^{-6} per g. of material	Retention times: Carbowax 5	Apiezon 4	1,2,3-Tris-cyanopropane 58	QF-1 61	DEGA 60	Ucon 56a	Triton X-305	Silicone Grease	QF-1	Mass Spec. Verification
Acetaldehyde	P	< 130	3.8	1.30						1.55		43, 29, 42
Acetone	P	< 68	5.1	NC	12.5			3.73				
Acrolein	M		5.4	1.70						NC		
Benzene	P	< 28	7.7	NC	NC		13.02	7.84				78, 77, 76, 52, 51, 50, 39, 38, 37
Butane	P	2400	2.10	1.50						1.47		
Chloroform	M	2300	10.1	3.3	12.65					NC		
2-Chloropropane	M		3.1	2.09	2.9					NC		
Cyclohexane	M		3.1	NC	2.2			3.73				57, 56, 55
Cis-1,2-Dichloroethylene	P	< 28			12.5?			8.36	2.12	3.90		
1,2-Dichloroethane	M		12.0	NC	NC	3.00						
1,1-Dimethylcyclohexane	M		NC	13.6	3.2							
_n_-Decane	P		NC	NC	NC	7.8	14.50		3.63	24.16	9.05	
Ethanol	D	118	NC	1.70	14.7	1.60				NC		46, 45, 31
Ethylbenzene	M		18.1	NC	NC					15.50		93, 92, 91, 51, 50
_n_-Heptane	P	1550	3.0	5.6	1.5					8.26		
2-Heptene	M		3.1	5.6	2.2					10.3?		
Methanol	P	2100	7.7	1.30	12.5	1.60?				1.47		
Methylcyclohexane	D	583	3.8	7.6	2.5	2.32						
_n_-Octane	P	<1300	3.8	NC	2.2	3.40						
1-Propanol	M							10.75	2.69	NC		
Tetrachloroethylene	M		10.1	NC	NC					13.78		
Toluene	D	2125	12.0	NC	NC		11.30			10.30		93, 92, 91, 51, 50
1,1,1-Trichloroethane	P	2310	5.4	3.9	6.8							
m-Xylene	M		18.1	NC	NC			20.8				

7.5 INFRARED SPECTRA

Tables XIV-XV summarize infrared spectra (10 meter gas cell) on the exposure atmospheres of solder lug insulation (item #10) and Tygon #1 (item #1). Infared spectra with the long path cell on the Min-K-1301 (item #3), Teflon wire insulation (item #7), insulation blanket (item #8), and sponge insulation bonded with RTV 102 (item #9) exposures were also obtained but they were so dilute that they showed no significant absorptions.

Infrared spectra were also obtained on all 10 exposures using short (0.1 meter) gas cells; all these showed no significant absorptions.

The spectra from two samples with high enough concentrations of gaseous materials to show significant absorption bands were not distinctive enough to identify any new compounds or any of the compounds whose identification was doubtful. Because of the large number of compounds present and their low concentration the only information that could be obtained from the infrared spectra was verification of previously identified compounds.

TABLE XIV

INFRARED SPECTRUM OF ATMOSPHERE FROM TYGON (#1) TEST

Absorption, cm^{-1}	Indication of	Verification of compounds found by GLC
3700	Hydroxy groups	Methanol, ethanol
3600	Hydroxy groups	1-butanol 2-propanol
2900	Aliphatic hydrogen	
2710, 2810 weak	$-\overset{\overset{\textstyle O}{\parallel}}{C}-H$ group	Acetaldehyde, butyraldehyde
2310	Carbon dioxide	
1725 strong, broad	Ketones, esters, aldehydes	Ethyl and methyl acetates, acetone, methyl ethyl ketone, acetaldehyde, butyraldehyde
1515		
1365	Methyl groups bonded to carbon atoms	Acetone, methyl ethyl ketone, methylcyclohexane, *n*-alkanes
1210	Ketones, esters	Ethyl and methyl acetates, acetone, methyl ethyl ketone

TABLE XV

INFRARED SPECTRUM OF ATMOSPHERE FROM SOLDER LUG INSULATION

Absorption, cm^{-1}	Indication of	Verification of compounds found by GLC
3700	Hydroxy groups	Methanol, ethanol
3090	Vinyl hydrogen	Acrolein, 2-heptene, *cis*-1,2-dichloro-ethylene
2900	Aliphatic hydrogen	
2800	$-\overset{O}{\overset{\|}{C}}-H$	Acetaldehyde, acrolein
2310	Carbon dioxide	
1730	Ketones, aldehydes	
1510		

7.6 QUANTITATIVE CALCULATIONS

The responses on the gas chromatograph were calibrated in the equivalents of peak height (at 4x attenuation) for milligrams of material. For all quantitative data 5 ml. (at 760 6 mm pressure) injections were used. The corrected volume Vc at 1 atm and 25°C is

$$V_c = \frac{V \cdot P_f}{P_R} = \text{6th column (\underline{a} series of Quantitative Tables XVI-XXV)}$$

Where V = volume of container in milliliters (5th column, a series of Quantitative Tables XVI-XXV)

P_f = Pressure of test vessel when sampling occurred

P_R = Room pressure at time of injection. (It was always 754-763.5 mm).

Fourth column, a series Tables on Quantitative Analysis is $\frac{P_f}{P_R}$

Since

PH/4 - Peak height at 4 times attenuation as read from the chromatograph (1st column in the a series of Tables).

CF = Calibration factor determined from calibration charts for PH/4

The total amount (in mg) of material in a 5 ml injection is (PH/4)·CF and this is given in the second column in the a series of Tables.

The total amount of material given off (Wm) is that found in the 5 ml aliquot multiplied by the total corrected volume (Vc) and divided by 5 or

$$Wm = \frac{Vc}{5}(PH/4)(CF)$$

Substituting:

$$Wm = \frac{V \cdot P_f}{5\ P_R}(PH/4)(CF)$$

The milligrams per gram (3rd column, b series of Quantitative Tables) of material were then obtained by dividing W_m (6th column, a series, or 2nd column, b series) by the total weight (1st column, b series) of material used for the exposure.

For some of the compounds tentatively identified by retention times there is no estimation of quantities present. This is due to one or more of the following factors: (1) The certainty of identification or amount of material present did not warrant quantitative calibrations for that compound. (2) The amount of material present was so small quantitative determination was impossible even though quantitative calibration curves were available for the compound in question. (3) Large amounts of other materials with similar retention times on the columns used for quantitative analysis made determination of quantities present impossible.

7.7 WEIGHT BALANCE DATA AND DISCUSSION

The small weight losses shown in Table XXVI verify the conclusions from the DTA data that there would not be significant weight loss from the samples exposed at 200^{o}. Examination of the material revealed no drastic changes such as charring. (Compare with 9.0 Series C tests). The two samples of Tygon were the only materials that underwent an observed change at all. Both of these materials were considerably less compliant after the Series A tests. This indicated that there had been a significant loss or modification of the plasticizer.

7.8 ORIGINAL DATA

Copies of the raw data (GLC curves, calibration data, and mass spectra) from which all this information was obtained have been submitted to the appropriate personnel at NASA Langley.

TABLE XVIa

CALCULATION OF QUANTITIES EVOLVED FROM TYGON, #1

		Sample #1						Sample #2						Sample #3					
		1	2	3	4	5	6	1	2	3	4	5	6	1	2	3	4	5	6
Compound	Col. No.	Ph/4	mg/5cc @ atm	$Pf_{(mm)}$	Pf/760	Vol.	mg_T	Ph/4	mg/5cc	$Pf_{(mm)}$	Pf/760	Vol.	mg_T	Ph/4	mg/5cc	$Pf_{(mm)}$	Pf/760	Vol.	mg_T
			$\times 10^{-5}$				$\times 10^{-5}$		$\times 10^{-5}$				$\times 10^{-5}$		$\times 10^{-5}$				$\times 10^{-5}$
Acetaldehyde	5	476	15.4	995.1	1.3093	2010	8105	596	22.4	969.6	1.2758	2010	11488	1240	47.6	963.7	1.2680	2010	24264
Acetone	58	228	32	"	"	"	16843	668	250	"	"	"	128217	306	44.5	"	"	"	22683
Benzene	58	228	19.6	"	"	"	10316	668	50.0	"	"	"	25644	306	27.0	"	"	"	13763
Carbon tetra-chloride	58	12.7	13.6	"	"	"	7158	328	348	"	"	"	178479	95.4	104	"	"	"	53013
Cyclohexane	4	107	34	"	"	"	17895	34	1.14	"	"	"	585						
Ethanol	5	49.5	3.06	"	"	"	1610	388	23.8	"	"	"	12206	76.6	4.2	"	"	"	2141
Ether	58	75.7	2.1	"	"	"	1105	176	6.0	"	"	"	3077	73.6	2.0	"	"	"	1019
Heptane	58	67	1.12	"	"	"	589	216	2.85	"	"	"	1462	88.8	1.52	"	"	"	775
Hexane	58	79	1.09	"	"	"	574	224	3.82	"	"	"	1959	114	1.96	"	"	"	999
Methyl acetate	58	1.5	<0.02	"	"	"	<10.5							21	2.08	"	"	"	1060
Methylcyclo-hexane	58	156	4.34	"	"	"	2284							100	2.78	"	"	"	1417
Methylene chloride	58	5	2.1	"	"	"	1105	3	1.0	"	"	"	53						
Methyl ethyl ketone	58	22.3	2.72	"	"	"	1431	80.2	9.5	"	"	"	4872	29.5	3.60	"	"	"	1835
Octane	58	75.7	2.1	"	"	"	1105	176	6.0	"	"	"	3077	73.6	2.0	"	"	"	1019
Pentane	58	419	3.0	"	"	"	1579	1320	11.7	"	"	"	6001	459	3.45	"	"	"	1759
2-Propanol	5	50.2	5.1	"	"	"	2684	388	34	"	"	"	17438	98.7	10.7	"	"	"	5454
Toluene	5	2.0	0.34	"	"	"	179	6.7	0.70	"	"	"	359	1.4	0.30	"	"	"	153

TABLE XVIb

CALCULATION OF QUANTITIES EVOLVED FROM TYGON, #1

Compound	1 g_1	2 mg_1	3 mg/g	1 g_2	2 mg_2	3 mg/g	1 g_3	2 mg_3	3 mg/g	Average
		$\times 10^{-5}$	10^{-5}		$\times 10^{-5}$	10^{-5}		$\times 10^{-5}$	10^{-5}	mg/g $\times 10^{-6}$
Acetaldehyde	20.8489	8105	388.8	19.7263	11,488	582	20.0629	24,264	1209	<7266
Acetone	"	16,843	807	"	128,217	6499	"	22,683	1130	[a]28,120
Benzene	"	10,316	494.8	"	25,644	1046	"	13,763	686	[b]7423
Carbon tetrachloride	"	7158	343	"	178,479	9047	"	53,013	2642	[c]3430
Cyclohexane	"	17,895	858	"	585	29.7	"			2959
Diethyl ether	"	1105	53	"	3077	156	"	1019	50.8	866
Ethanol	"	1610	77.2	"	12,206	618.8	"	2141	106	2673
Heptane	"	589	28.2	"	1462	74.1	"	775	38.6	469
Hexane	"	574	27.5	"	1959	99.3	"	999	49.8	589
Methyl acetate	"	<10.5	<0.50					1060	52.8	<530
Methylcyclohexane	"	2284	110				"	1417	71	90
Methylethyl ketone	"	1431	68.6	"	4872	246	"	1835	91.5	1354
Methylene chloride	"	1105	53	"	513	26				40
Octane	"	679	32.6	"	1590	80.6	"	637	31.7	483
Pentane	"	1579	3.0	"	6001	304	"	1759	87.7	1316
2-Propanol	"	2684	128.7	"	17,438	884	"	5454	272	428
Toluene	"	179	8.6	"	359	18.2	"	153	7.6	11.5

[a]Peak contains at least 10 per cent benzene.

[b]Peak contains at least 60 per cent acetone.

[c]Lowest single value since high readings were probably due to an artifact; see text.

TABLE XVIIa

CALCULATION OF QUANTITIES EVOLVED FROM TYGON #2

		Sample #1						Sample #2						Sample #3					
		1	2	3	4	5	6	1	2	3	4	5	6	1	2	3	4	5	6
Compound	Col. No.	Ph/4	mg/5cc @ atm	Pf mm	Pf/760	Vol.	Total mg	Ph/4	mg/5cc @ atm	Pf mm	Pf/760	Vol.	Total mg	Ph/4	mg/5cc @ atm	Pf mm	Pf/760	Vol.	Total mg
Acetone																			
Carbon tetra-chloride	58	50	54.0×10^{-5}	965	1.2697	2018	27672×10^{-5}	10	10.8×10^{-5}	1067	1.4039	2018	6119×10^{-5}	20	21.5×10^{-5}	964	1.2684	2010	10963×10^{-5}
Cyclohexane	5	-	$<0.02 \times 10^{-5}$	"	"	"	$<10 \times 10^{-5}$	-	$<0.02 \times 10^{-5}$	"	"	"	$<11 \times 10^{-5}$	-	$<0.02 \times 10^{-5}$	"	"	"	$<10 \times 10^{-5}$
Diethyl ether	58	30	1.09×10^{-5}	"	"	"	558×10^{-5}	56	NC					46	1.70×10^{-5}	"	"	"	867×10^{-5}
Ethanol	5	16	0.95×10^{-5}	"	"	"	487×10^{-5}	40	2.47×10^{-5}	"	"	"	1400×10^{-5}	56	3.47×10^{-5}	"	"	"	1769×10^{-5}
n-Heptane	58	38	0.59×10^{-5}	"	"	"	302×10^{-5}	60	0.99×10^{-5}	"	"	"	561×10^{-5}	72	1.21×10^{-5}	"	"	"	617×10^{-5}
n-Hexane		68	0.82×10^{-5}	"	"	"	420×10^{-5}	89	1.34×10^{-5}	"	"	"	759×10^{-5}	85	1.24×10^{-5}	"	"	"	632×10^{-5}
Methyl acetate	58	1	$<0.1 \times 10^{-5}$	"	"	"	$<51 \times 10^{-5}$	1	$<0.1 \times 10^{-5}$	"	"	"	$<57 \times 10^{-5}$	1	$<0.1 \times 10^{-5}$	"	"	"	$<51 \times 10^{-5}$
Methylcyclo-hexane	58	68	1.89×10^{-5}	"	"	"	968×10^{-5}	89	2.48×10^{-5}	"	"	"	1405×10^{-5}	81	2.26×10^{-5}	"	"	"	1152×10^{-5}
Methylene chloride	58	4	1.6×10^{-5}	"	"	"	820×10^{-5}	4	1.6×10^{-5}	"	"	"	906×10^{-5}	8	3.9×10^{-5}	"	"	"	1988×10^{-5}
Methyl ethyl ketone	5	22	0.88×10^{-5}	"	"	"	451×10^{-5}	72	2.74×10^{-5}	"	"	"	1552×10^{-5}	72	2.74×10^{-5}	"	"	"	1397×10^{-5}
	58	18	2.19×10^{-5}	"	"	"	1122×10^{-5}	28	3.42×10^{-5}	"	"	"	1938×10^{-5}	20	2.44×10^{-5}	"	"	"	1244×10^{-5}
n-Octane	58	30	0.52×10^{-5}	"	"	"	266×10^{-5}	48	0.82×10^{-5}	"	"	"	465×10^{-5}	32	0.55×10^{-5}	"	"	"	280×10^{-5}
n-Pentane	58	8x67	4.85×10^{-5}	"	"	"	2485×10^{-5}	8x71	5.15×10^{-5}	"	"	"	2918×10^{-5}	8x77	5.65×10^{-5}	"	"	"	2881×10^{-5}
2-Propanol	5	16	1.05×10^{-5}	"	"	"	538×10^{-5}	40	3.85×10^{-5}	"	"	"	2181×10^{-5}	56	5.75×10^{-5}	"	"	"	2932×10^{-5}
Toluene	5	-	$<0.3 \times 10^{-5}$	"	"	"	$<154 \times 10^{-5}$	-	$<0.3 \times 10^{-5}$	"	"	"	$<170 \times 10^{-5}$	1	$<0.3 \times 10^{-5}$	"	"	"	$<153 \times 10^{-5}$

TABLE XVIIb

CALCULATION OF QUANTITIES EVOLVED FROM TYCON, #2

Compound	1 g_1	2 mg_2	3 mg/g	1 g_2	2 mg_2	3 mg/g	1 g_3	2 mg_3	3 mg/g	Average
Acetone	19.5965	0.06175 0.01122	3151×10^{-6} 572×10^{-6}	18.4905	0.13825 0.01938	7477×10^{-6} 1048×10^{-6}	20.2524	0.22027 0.01244	$10{,}876 \times 10^{-6}$ 614×10^{-6}	[a] 7168×10^{-6} [b] 745×10^{-6}
Carbon tetrachloride	"	0.27672	$14{,}121 \times 10^{-6}$	"	0.06119	3309×10^{-6}	"	0.10963	5413×10^{-6}	[c] 3860×10^{-6}
Cyclohexane	"	<0.00010	$<5 \times 10^{-6}$	"	<0.00011	$<6 \times 10^{-6}$	"	<0.00010	$<5 \times 10^{-6}$	$<6 \times 10^{-6}$
Diethyl ether	"	0.00558	285×10^{-6}	"			"	0.00867	428×10^{-6}	356×10^{-6}
Ethanol	"	0.00487	248×10^{-6}	"	0.01400	757×10^{-6}	"	0.01769	873×10^{-6}	[b] 626×10^{-6}
n-Heptane	"	0.00302	154×10^{-6}	"	0.00561	303×10^{-6}	"	0.00617	305×10^{-6}	254×10^{-6}
n-Hexane	"	0.00420	214×10^{-6}	"	0.00759	410×10^{-6}	"	0.00632	312×10^{-6}	312×10^{-6}
Methyl acetate	"	<0.00051	$<26 \times 10^{-6}$	"	<0.00057	$<31 \times 10^{-6}$	"	<0.00051	$<0.25 \times 10^{-6}$	$<31 \times 10^{-6}$
Methylcyclohexane	"	0.00985	503×10^{-6}	"	0.01405	760×10^{-6}	"	0.01152	569×10^{-6}	611×10^{-6}
Methylene chloride	"	0.00820	418×10^{-6}	"	0.00906	490×10^{-6}	"	0.01988	982×10^{-6}	680×10^{-6}
Methyl ethyl ketone	"	0.00451	230×10^{-6}	"	0.01552	839×10^{-6}	"	0.01397	690×10^{-6}	586×10^{-6}
n-Octane	"	0.00266	136×10^{-6}	"	0.00465	252×10^{-6}	"	0.00280	138×10^{-6}	[a] 175×10^{-6}
n-Pentane	"	0.02485	1268×10^{-6}	"	0.02918	1578×10^{-6}	"	0.02881	1422×10^{-6}	1423×10^{-6}
2-Propanol	"	0.00538	274×10^{-6}	"	0.02181	1180×10^{-6}	"	0.02932	1448×10^{-6}	[d] 967×10^{-6}
Toluene	"	<0.00154	$<78 \times 10^{-6}$	"	<0.00170	$<92 \times 10^{-6}$	"	<0.00153	$<76 \times 10^{-6}$	$<92 \times 10^{-6}$

[a] Includes some carbon tetrachloride .

[b] Includes some 2-propanol.

[c] Average of two with lower concentration, or the higher concentration was probably an artifact; see text.

[d] Includes some ethanol.

TABLE XVIIIa

Calculation of Quantities Evolved from Min-k Fiber, #3

		Sample #1						Sample #2						Sample #3					
		1	2	3	4	5	6	1	2	3	4	5	6	1	2	3	4	5	6
Compound	Col. No.	PH/4	mg/5cc @ atm	Pf mm	Pf/760	Vol.	mg_t	PH/4	mg/5cc @ atm	Pf mm	Pf/760	Vol.	mg_T	PH/4	mg/5cc @ atm	Pf mm	Pf/760	Vol.	mg
Acetaldehyde	5	2	$<0.1 \times 10^{-5}$	966	1.2710	2003	$< 51\times10^{-5}$	3	$<0.1\times10^{-5}$	1026	1.3500	2003	$<54\times10^{-5}$	4	$<0.1\times10^{-3}$	964	1.2684	2018	$<51\times10^{-5}$
Acetone	5	8	0.52×10^{-5}	"	"	"			0.86×10^{-5}	"	"	"		17	1.13×10^{-5}	"	"	"	563×10^{-5}
Benzene	58	2	0.07×10^{-5}	"	"	"	36×10^{-5}	2	0.07×10^{-5}	"	"	"	38×10^{-5}	9	0.7×10^{-5}	"	"	"	358×10^{-5}
Butane	58							2x55	0.95×10^{-5}	"	"	"	514×10^{-5}	72.5x4	2.55×10^{-5}	"	"	"	1305×10^{-5}
Diethyl ether	58	--	$<0101\times10^{-5}$	"	"	"	$< 5\times10^{-5}$	--	$<0.01\times10^{-5}$	"	"	"	$< 5\times10^{-5}$	1	$<0.01\times10^{-5}$	"	"	"	$< 5\times10^{-5}$
Ethanol	5	3	012×10^{-5}	"	"	"	61×10^{-5}	3	0.12×10^{-5}	"	"	"	65×10^{-5}	10	0.57×10^{-5}	"	"	"	292×10^{-5}
formaldehyde	5	--	$<0.1\times10^{-5}$	"	"	"	$<51\times10^{-5}$	3	0.2×10^{-5}	"	"	"	108×10^{-5}	--	$<0.1\times10^{-5}$	"	"	"	251×10^{-5}
n-Hexane	5		$<0.01\times10^{-5}$	"	"	"	$<5\times10^{-5}$	1	$<0.01\times10^{-5}$	"	"	"	$<5\times10^{-5}$	--	$<0.01\times10^{-5}$	"	"	"	251×10^{-5}
Methanol	5	--	$<0.05\times10^{-5}$	"	"	"	$<36\times10^{-5}$	5	0.3×10^{-5}	"	"	"	162×10^{-5}	--	$<0.05\times10^{-5}$	"	"	"	$<26\times10^{-5}$
Methyl acetate	58	--	$<0.1\times10^{-5}$	"	"	"	$<51\times10^{-5}$	--	$<0.1\times10^{-5}$	"	"	"	$<54\times10^{-5}$	--	$<0.1\times10^{-5}$	"	"	"	$<51\times10^{-5}$
Methylene chloride	58	--	$<0.1\times10^{-5}$	"	"	"	$<51\times10^{-5}$	--	$<0.1\times10^{-5}$	"	"	"	$<54\times10^{-5}$	--	$<0.1\times10^{-5}$	"	"	"	$<51\times10^{-5}$
n-Pentane	58	77	0.64×10^{-5}	"	"	"	326×10^{-5}	63	0.51×10^{-5}	"	"	"	276×10^{-5}	116	1.0×10^{-5}	"	"	"	517×10^{-5}
Toluene	5	--	$<0.3 \times10^{-5}$	966	1.2710	2003	$<153\times10^{-5}$	--	$<0.3 \times10^{-5}$	"	"	"	$<162\times10^{-5}$	2	0.34×10^{-5}	"	"	"	

TABLE XVIIIb

CALCULATION OF QUANTITIES EVOLVED FROM MIN-K, #3

Compound	[1] g_1	[2] mg_1	[3] mg/g	[1] g_2	[2] mg_2	[3] mg/g	[1] g_3	[2] mg_3	[3] mg/g	Average
Acetaldehyde	5.0459	<0.00051	$<101 \times 10^{-6}$	4.9199	<0.00054	$<110 \times 10^{-6}$	5.2928	<0.00051	$<96 \times 10^{-6}$	$<110 \times 10^{-6}$
Acetone	"	<0.00025	$<50 \times 10^{-6}$	"	<0.00027	$<55 \times 10^{-6}$	"	0.00563	1064×10^{-6}	[a] $<1064 \times 10^{-6}$
Benzene	"	0.00036	71×10^{-6}	"	0.00038	77×10^{-6}	"	0.00358	676×10^{-6}	275×10^{-6}
n-Butane	"			"	0.00514	1045×10^{-6}	"	0.01305	2466×10^{-6}	1756×10^{-6}
Ethanol	"	0.00061	121×10^{-6}	"	0.00065	132×10^{-6}	"	0.00292	552×10^{-6}	268×10^{-6}
Ether	"	<0.00005	$<10 \times 10^{-6}$	"	<0.00005	$<10 \times 10^{-6}$	"	<0.00005	$<10 \times 10^{-6}$	$<10 \times 10^{-6}$
Formaldehyde	"	<0.00051	$<101 \times 10^{-6}$	"	0.00108	220×10^{-6}	"	<0.00051	$<96 \times 10^{-6}$	$<220 \times 10^{-6}$
n-Hexane	"	<0.00005	$<10 \times 10^{-6}$	"	<0.00005	$<10 \times 10^{-6}$	"	<0.00005	$<10 \times 10^{-6}$	$<10 \times 10^{-6}$
Methanol	"	<0.00036	$<71 \times 10^{-6}$	"	0.00162	329×10^{-6}	"	0.00026	49×10^{-6}	$<329 \times 10^{-6}$
Methyl acetate	"	<0.00051	$<101 \times 10^{-6}$	"	<0.00054	$<110 \times 10^{-6}$	"	<0.00051	$<96 \times 10^{-6}$	$<110 \times 10^{-6}$
Methylene chloride	"	<0.00051	$<101 \times 10^{-6}$	"	<0.00054	$<110 \times 10^{-6}$	"	<0.00051	$<96 \times 10^{-6}$	$<110 \times 10^{-6}$
n-Pentane	"	0.00326	646×10^{-6}	"	0.00276	561×10^{-6}	"	0.00517	977×10^{-6}	[b] 728×10^{-6}
Toluene	"	<0.00153	$<303 \times 10^{-6}$	"	<0.00162	$<329 \times 10^{-6}$	"	0.00174	329×10^{-6}	$<329 \times 10^{-6}$

[a] Includes equivalent of 275×10^{-6} mg/g of benzene.

[b] Includes minor amounts (less than 10 per cent) of other compounds, probably hydrocarbons.

TABLE XIXa

CALCULATION OF QUANTITIES EVOLVED FROM SILICONE COATED GLASS CLOTH, #4

		Sample #1						Sample #2						Sample 3					
		1	2	3	4	5	6	1	2	3	4	5	6	1	2	3	4	5	6
Compound	Col. No.	Ph/4	$\frac{mg}{5cc}$ @ atm	Pfmm	$\frac{Pf}{760}$	Vol.	Total mg	Ph/4	$\frac{mg}{5cc}$ @ atm	Pfmm	$\frac{Pf}{760}$	Vol.	Total mg	Ph/4	$\frac{mg}{5cc}$ @ atm	Pfmm	$\frac{Pf}{760}$	Vol.	Total mg
Acetone	58	31	4.5×10^{-5}	1017	1.3381	2003	24.2×10^{-5}	26	3.75×10^{-5}	1017	1.3381	2010	2017×10^{-5}	23	3.25×10^{-5}	964	1.2684	2010	1657×10^{-5}
	5	17	1.14×10^{-5}	"	"	"	611×10^{-5}	20	1.34×10^{-5}	"	"	"	721×10^{-5}	59	3.98×10^{-5}	"	"	"	2029×10^{-5}
Benzene	58	31	2.72×10^{-5}	"	"	"	1458×10^{-5}	26	2.26×10^{-5}	"	"	"	1216×10^{-5}	23	2.0×10^{-6}	"	"	"	1020×10^{-5}
Carbon tetra-chloride	58	1	$<1 \times 10^{-5}$	"	"	"	$<536 \times 10^{-5}$	1	$<1 \times 10^{-5}$	"	"	"	$<538 \times 10^{-5}$	2	1.6×10^{-5}	"	"	"	816×10^{-5}
Ethanol	5	13	0.76×10^{-5}	"	"	"	407×10^{-5}	11	0.64×10^{-6}	"	"	"	344×10^{-5}	11	0.64×10^{-6}	"	"	"	326×10^{-5}
Diethyl ether	58	1	$<0.01 \times 10^{-5}$	"	"	"	$<5 \times 10^{-5}$	2.5	0.02×10^{-5}	"	"	"	11×10^{-5}	3	0.04×10^{-5}	"	"	"	20×10^{-5}
Ethyl formate	58	-	$<0.4 \times 10^{-5}$	"	"	"	$<214 \times 10^{-5}$	-	$<0.4 \times 10^{-5}$	"	"	"	$<215 \times 10^{-5}$	1	$<0.4 \times 10^{-5}$	"	"	"	$<204 \times 10^{-5}$
n-Hexane	58	5	$<0.01 \times 10^{-5}$	"	"	"	$<5 \times 10^{-5}$	9	$<0.01 \times 10^{-5}$	"	"	"	$<5 \times 10^{-5}$	-	$<0.01 \times 10^{-5}$	"	"	"	$<5 \times 10^{-5}$
	5	-	$<0.01 \times 10^{-5}$	"	"	"	$<5 \times 10^{-5}$	3	$<0.01 \times 10^{-5}$	"	"	"	$<5 \times 10^{-5}$	1	$<0.01 \times 10^{-5}$	"	"	"	$<5 \times 10^{-5}$
Methanol	5	-	$<0.05 \times 10^{-5}$	"	"	"	$<27 \times 10^{-5}$	-	$<0.05 \times 10^{-5}$	"	"	"	$<27 \times 10^{-5}$	-	$<0.05 \times 10^{-5}$	"	"	"	$<25 \times 10^{-5}$
n-Octane	58	1	$<0.05 \times 10^{-5}$	"	"	"	$<27 \times 10^{-5}$	2.5	0.06×10^{-5}	"	"	"	32×10^{-5}	3	0.07×10^{-5}	"	"	"	36×10^{-5}
n-Pentane	58	10	$<0.01 \times 10^{-5}$	"	"	"	$<5 \times 10^{-5}$	12	0.02×10^{-5}	"	"	"	11×10^{-5}	50×8	3.6×10^{-5}	"	"	"	1836×10^{-5}

TABLE XIXb

CALCULATION OF QUANTITIES EVOLVED FROM SILICONE COATED GLASS CLOTH, #4

Compound	1 g_1	2 mg_1	3 mg/g	1 g_2	2 mg_2	3 mg/g	1 g_3	2 mg_3	3 mg/g	Average
Acetone	30.1853	0.02412 0.00611	799×10^{-6} 202×10^{-6}	29.4242	0.02017 0.00721	685×10^{-6} 245×10^{-6}	29.6989	0.01657 0.02029	558×10^{-6} 683×10^{-6}	681×10^{-6} [a] 377×10^{-6} [b]
Benzene	"	0.01458	483×10^{-6}	"	0.01216	413×10^{-6}	"	0.01020	343×10^{-6}	413×10^{-6} [c]
Carbon tetrachloride	"	<0.00536	$<178 \times 10^{-6}$	"	<0.00538	$<183 \times 10^{-6}$	"	0.00816	275×10^{-6}	$<275 \times 10^{-6}$
Diethyl ether	"	<0.00005	$<2 \times 10^{-6}$	"	0.00011	4×10^{-6}	"	0.00020	7×10^{-6}	$<7 \times 10^{-6}$ [d]
Ethanol	"	0.00407	135×10^{-6}	"	0.00344	117×10^{-6}	"	0.00326	110×10^{-6}	121×10^{-6}
Ethyl formate	"	<0.00214	$<71 \times 10^{-6}$	"	<0.00215	$<73 \times 10^{-6}$	"	<0.00204	$<69 \times 10^{-6}$	$<73 \times 10^{-6}$ [e]
_n_-Hexane	"	<0.00005	$<2 \times 10^{-6}$	"	<0.00005	$<2 \times 10^{-6}$	"	<0.00005	$<2 \times 10^{-6}$	$<2 \times 10^{-6}$
Methanol	"	<0.00027	$<9 \times 10^{-6}$	"	<0.00027	$<9 \times 10^{-6}$	"	<0.00025	$<8 \times 10^{-6}$	$<9 \times 10^{-6}$
_n_-Octane	"	<0.00027	$<9 \times 10^{-6}$	"	0.00032	11×10^{-6}	"	0.00036	12×10^{-6}	$<12 \times 10^{-6}$
_n_-Pentane	"	<0.00027	$<9 \times 10^{-6}$	"	0.00032	11×10^{-6}	"	0.00036	12×10^{-6}	$<12 \times 10^{-6}$

[a] Includes at least 1/3 benzene.

[b] Includes equivalent of less than 275×10^{-6} mg/g of carbon tetrachloride.

[c] Includes at least 1/3 acetone.

[d] Includes at least 1/3 _n_-octane.

[e] Much less than quantity shown because of overlap with ethanol.

TABLE XXa

CALCULATION OF QUANTITIES EVOLVED FROM COHRLASTIC, #5

		Sample #1						Sample #2						Sample #3					
		1	2	3	4	5	6	1	2	3	4	5	6	1	2	3	4	5	6
Compound	Col. No.	Ph/4	mg/5cc @ atm $\times 10^{-5}$	Pfmm	Pfmm/atm	Vol. cc Container	Total mg $\times 10^{-5}$	Ph/4	mg/5cc @ atm	Pfmm	Pfmm/atm	Vol. cc Container	Total mg $\times 10^{-5}$	Ph/4	mg/5cc @ atm $\times 10^{-5}$	Pfmm	Pfmm/atm	Vol. cc Container	Total mg $\times 10^{-5}$
Acetaldehyde	5								2.16×10^{-5}	1151	1.514	2018	1647		2.24	964	1.268	2010	1142
Acetone	5							31	$2.08 \times 10^{-5}_{c}$ CCl_4	"	"	"	1270.9	40.5	2.72	"	"	"	1386.4
	58	40 $\bar{c}$ MeOH	5.9 MeOH	971	1.277	2.003	3018.2	22	$3.1 \times 10^{-5}_{c}$ MeOH	"	"	"	1894.2	43	6.4	"	"	"	3262.3
	58	-	<0.1	"	"	"	<511	-	$<0.1 \times 10^{-5}$	"	"	"	<61	-	<0.1	"	"	"	<51
Butane	58	-	<0.05	"	"	"	<25.5	-	$<0.05 \times 10^{-5}$	"	"	"	<30.5	-	<0.05	"	"	"	<25.4
Carbon tetrachloride	58	6	0.1	"	"	"	51.1	4	0.05×10^{-5}	"	"	"	30.5	14	0.34	"	"	"	173.3
Ethanol	5							58.5 × 16	218×10^{-5}	"	"	"	133208	73 × 16	275	"	"	"	140178.5
Formaldehyde	5							58	3.1×10^{-5}	"	"	"	1894.2	59.5	2.48	"	"	"	1264.1
Ethyl formate	58	3	0.5	"	"	"	256	2	0.4×10^{-5}	"	"	"	244	3	0.5	"	"	"	255
Methanol	5								$<0.05 \times 10^{-5}$	"	"	"	<30.5		<0.05	"	"	"	<25.4
Methyl ethyl ketone	5	-	<0.05					-	$<0.10 \times 10^{-5}$	"	"	"	<61.1	-	<0.10	"	"	"	<50.9
Methylene chloride	58	3.5	1.3	"	"	"	666	2	0.5×10^{-5}	"	"	"	305.5	2	0.5	"	"	"	254.8
n-Pentane	58	-	<0.05	"	"	"	<25.5	-	$<0.05 \times 10^{-5}$	"	"	"	<30.5	-	<0.05	"	"	"	<25.4

TABLE XXb

CALCULATION OF QUANTITIES EVOLVED FROM COHRLASTIC, #5

Compound	1 g_1	2 mg_1	3 mg/g	1 g_2	2 mg_2	3 mg/g	1 g_3	2 mg_3	3 mg/g	Average
Acetaldehyde	27.2296			28.3902	0.01647	580×10^{-6}	28.3615	0.01142	403×10^{-6}	492×10^{-6}[a]
Acetone	"	0.03018	1108.4×10^{-6}	"	0.01271 0.01894	447.7×10^{-6} 667.1×10^{-6}	"	0.01386 0.03262	488.7×10^{-6} 1150×10^{-6}	468×10^{-6}[b] 975×10^{-6}[c]
Butane	"	<0.00026	$<9.5 \times 10^{-6}$	"	<0.00030	$<10.6 \times 10^{-6}$	"	<0.00025	$<8.8 \times 10^{-6}$	$<11 \times 10^{-6}$
Carbon tetrachloride				"	0.00978	344.5×10^{-6}	"	0.00816	287.7×10^{-6}	316×10^{-6}[d]
Ethanol				"	1.33208	0.04692	"	1.40178	0.04942	$48,170 \times 10^{-6}$
Ethyl formate	"	0.00256	94×10^{-6}	"	0.00244	86×10^{-6}	"	0.00255	90×10^{-6}	90×10^{-6}[e]
Formaldehyde				"	0.01894	667.1×10^{-6}	"	0.01264	445.7×10^{-6}	556×10^{-6}[f]
Methanol				"	<0.00030	$<10.6 \times 10^{-6}$	"	<0.00025	$<8.8 \times 10^{-6}$	$<11 \times 10^{-6}$[g]
Methylcyclohexane	"	0.00051	18.7×10^{-6}	"	0.00030	10.6×10^{-6}	"	0.00173	61.0×10^{-6}	30×10^{-6}
Methylene chloride	"	0.00665	244.2×10^{-6}	"	0.00306	107.8×10^{-6}	"	0.00255	89.9×10^{-6}	147×10^{-6}[h]
Methyl ethyl ketone	"	<0.00051	$<18.7 \times 10^{-6}$	"	<0.00061	$<21.5 \times 10^{-6}$	"	<0.00051	$<18.0 \times 10^{-6}$	$<22 \times 10^{-6}$
n-Pentane	"	<0.00026	$<9.5 \times 10^{-6}$	"	<0.00030	$<10.6 \times 10^{-6}$	"	<0.00025	$<8.8 \times 10^{-6}$	$<11 \times 10^{-6}$

[a] Includes at least 20 per cent formaldehyde.

[b] Includes equivalent of approximately 300×10^{-6} mg of carbon tetrachloride.

[c] Includes a small amount of methanol.

[d] Includes a small amount of hexamethylcyclotrisiloxane.

[e] Includes at least 25 per cent methylene chloride.

[f] Includes at least 25 per cent formaldehyde.

[g] Includes a small amount of benzene.

[h] Includes a small amount of ethyl formate.

TABLE XXIa

CALCULATION OF QUANTITIES EVOLVED FROM ALUMINUM TAPE, #6

		Sample #1						Sample #2						Sample #3					
		1	2	3	4	5	6	1	2	3	4	5	6	1	2	3	4	5	6
Compound	Col. No.	Ph/4	$\frac{mg}{5cc}$ @ atm	Pf mm	$\frac{Pf}{760}$	Vol.	mg	Ph/4	$\frac{mg}{5cc}$ @ atm	Pf mm	$\frac{Pf}{760}$	Vol.	mg.	Ph/4	$\frac{mg}{5cc}$ @ atm	Pf mm	$\frac{Pf}{760}$	Vol.	mg
n-Butane	58	101	0.87×10^{-5}	760	1	2010	350×10^{-5}	192	1.72×10^{-5}	760	1	2003	689×10^{-5}	57	0.45×10^{-5}	961	1.2644	2018	230×10^{-5}
n-Pentane	58	46	0.34×10^{-5}	"	1	"	137×10^{-5}	26	0.15×10^{-5}	"	1	"	60×10^{-5}	22	0.12×10^{-5}	"	"	"	61×10^{-5}
	5	17	0.24×10^{-5}	"	1	"	96×10^{-5}	26	0.43×10^{-5}	"	1	"	172×10^{-5}	15	0.20×10^{-5}	"	"	"	102×10^{-5}
n-Hexane	58	50	0.37×10^{-5}	"	1	"	149×10^{-5}	11	$<0.01 \times 10^{-5}$	"	1	"	$< 4 \times 10^{-5}$	29	$<0.01 \times 10^{-5}$	"	"	"	$< 5 \times 10^{-5}$
Cyclopentane	58	31	0.47×10^{-5}	"	1	"	189×10^{-5}	29	0.44×10^{-5}	"	1	"	176×10^{-5}	20	0.26×10^{-5}	"	"	"	133×10^{-5}
Methylcyclo-pentane	58	29	0.35×10^{-5}	"	1	"	141×10^{-5}	32	0.37×10^{-5}	"	1	"	148×10^{-5}	21	0.14×10^{-5}	"	"	"	71×10^{-5}
Methylcyclo-hexane	5	20	0.45×10^{-5}	"	1	"	181×10^{-5}	56	1.27×10^{-5}	"	1	"	509×10^{-5}	27	0.61×10^{-5}	"	"	"	311×10^{-5}
	58	32	0.86×10^{-5}	"	1	"	346×10^{-5}	41	1.11×10^{-5}	"	1	"	445×10^{-5}	28	0.74×10^{-5}	"	"	"	378×10^{-5}
Carbon tetra-chloride	58	19	20.4×10^{-5}	"	1	"	820×10^{-5}	2	1.6×10^{-5}	"	1	"	641×10^{-5}	1.5	1.2×10^{-5}	"	"	"	612×10^{-5}
2-Propanol	5	2	2.95×10^{-5}	"	1	"	1186×10^{-5}	100	10.85×10^{-5}	"	1	"	4347×10^{-5}	46	4.55×10^{-5}	"	"	"	2322×10^{-5}
Acetone	5	41	2.76×10^{-5}	"	1	"		44	2.96×10^{-5}	"	1	"		30	2.0×10^{-5}	"	"	"	1021×10^{-5}
Methyl ethyl ketone	58	4	0.22×10^{-5}	"	1	"	88×10^{-5}	3	0.18×10^{-5}	"	1	"	72×10^{-5}	3	0.18×10^{-5}	"	"	"	92×10^{-5}
	5		0.17×10^{-5}	"	1	"	68×10^{-5}	7	0.32×10^{-5}	"	1	"	128×10^{-5}	6	0.28×10^{-5}	"	"	"	143×10^{-5}
Methyl acetate	58		$<0.1 \times 10^{-5}$	"	1	"	$<40 \times 10^{-5}$		$<0.1 \times 10^{-5}$	"	1	"	$<40 \times 10^{-5}$		$<0.1 \times 10^{-5}$	"	"	"	$<51 \times 10^{-5}$
n-Heptane	4	4	0.22×10^{-5}	"	1	"	88×10^{-5}		$<0.1 \times 10^{-5}$	"	1	"	$<40 \times 10^{-5}$	6	0.29×10^{-5}	"	"	"	148×10^{-5}
Toluene	5	2	0.34×10^{-5}	"	1	"	137×10^{-5}		$<0.3 \times 10^{-5}$	"	1	"	$<120 \times 10^{-5}$	4	0.50×10^{-5}	"	"	"	255×10^{-5}

TABLE XXIb

CALCULATION OF QUANTITIES EVOLVED FROM ALUMINUM TAPE, #6

Compound	1 g_1	2 mg_1	3 mg/g	1 g_2	2 mg_2	3 mg/g	1 g_3	2 mg_3	3 mg/g	Average
n-Butane	44.1224	0.00350	79×10^{-6}	50.2059	0.00689	137×10^{-6}	32.9041	0.00230	70×10^{-6}	95×10^{-6}
n-Pentane	"	0.00137	31×10^{-6}	"	0.00060	12×10^{-6}	"	0.00061	18×10^{-6}	20×10^{-6}
		0.00096	22×10^{-6}		0.00172	34×10^{-6}		0.00102	31×10^{-6}	29×10^{-6}
n-Hexane	"	0.00149	34×10^{-6}	"	<0.00004	$< 1 \times 10^{-6}$	"	<0.00005	$< 2 \times 10^{-6}$	$<34 \times 10^{-6}$
2-Heptene						at least $15 \cdot 10^{-6}$ mg/g estimated by diff.				
Cyclopentane	"	0.00189	43×10^{-6}	"	0.00176	35×10^{-6}	"	0.00133	40×10^{-6}	39×10^{-6}
Methylcyclopentane	"	0.00141	32×10^{-6}	"	0.00148	29×10^{-6}	"	0.00071	22×10^{-6}	28×10^{-6}
	"	0.00181	41×10^{-6}	"	0.00509	101×10^{-6}	"	0.00311	94×10^{-6}	[a] 79×10^{-6}
Methylcyclohexane	"	0.00346	78×10^{-6}	"	0.00445	89×10^{-6}	"	0.00378	115×10^{-6}	[b] 94×10^{-6}
Carbon tetrachloride	"	0.08201	1859×10^{-6}	"	0.00641	128×10^{-6}	"	0.00612	186×10^{-6}	724×10^{-6}
2-Propanol	"	0.01186	269×10^{-6}	"	0.04347	866×10^{-6}	"	0.02322	706×10^{-6}	614×10^{-6}
Acetone	"	0.01110	252×10^{-6}	"	0.01186	236×10^{-6}	"	0.01021	310×10^{-6}	[c] 266×10^{-6}
Methyl ethyl ketone	"	0.00088	20×10^{-6}	"	0.00072	14×10^{-6}	"	0.00092	28×10^{-6}	21×10^{-6}
Methyl acetate	"	< 0.00040	$< 9 \times 10^{-6}$	"	<0.00040	$< 8 \times 10^{-6}$	"	< 0.00051	$< 15 \times 10^{-6}$	$<15 \times 10^{-6}$
n-Heptane	"	0.00088	20×10^{-6}	"	<0.00040	$< 8 \times 10^{-6}$	"	0.00148	45×10^{-6}	[d] $<45 \times 10^{-6}$
Toluene	"	0.00137	31×10^{-6}	"	<0.00120	$< 24 \times 10^{-6}$	"	0.00255	77×10^{-6}	$<77 \times 10^{-6}$

[a] Peak includes a small undetermined amount of dimethyldimethoxysilane

[b] Peak includes at least equivalent of $15 \cdot 10^{-6}$ mg/g of 2-heptene

[c] Peak includes less than equivalent of $15 \cdot 10^{-6}$ mg/g of methyl acetate

[d] Peak includes at least equivalent of $15 \cdot 10^{-6}$ mg/g of 2-heptene

TABLE XXIIa

CALCULATION OF QUANTITIES EVOLVED FROM TEFLON WIRE INSULATION, #7

		Sample #1						Sample #2						Sample #3					
		1	2	3	4	5	6	1	2	3	4	5	6	1	2	3	4	5	6
Compound	Col. No.	PH/4	mg/5cc at atm	Pfmm	Pf/760	Vol.	mg_T	PH/4	mg/5cc at atm	Pfmm	Pf/760	Vol.	mg_T	PH/4	mg/5cc at atm	Pfmm	Pf/760	Vol.	mg_T
Acetaldehyde	5	6	$<0.1 \times 10^{-5}$	1121	1.4750	2010	$<59.2 \times 10^{-5}$	6	$<0.1 \times 10^{-5}$	1119	1.4723	2010	$<59.1 \times 10^{-5}$	7	$<0.1 \times 10^{-5}$	964	1.2684	2003	$<50.8 \times 10^{-5}$
Acetone	5		0.45×10^{-5}	"	"	"	266.8×10^{-5}	25	1.68×10^{-5}	"	"	"	994.3×10^{-5}	34	2.28×10^{-5}	"	"	"	1158.5×10^{-5}
Benzene	58		$<0.02 \times 10^{-5}$	"	"	"	$<11.8 \times 10^{-5}$		$<0.02 \times 10^{-5}$	"	"	"	$<11.8 \times 10^{-5}$		$<0.02 \times 10^{-5}$	"	"	"	$<10.1 \times 10^{-5}$
Chloroform	5		$<1.5 \times 10^{-5}$	"	"	"	$<889.4 \times 10^{-5}$												
Ethanol	5	2	0.06×10^{-5}	"	"	"	35.5×10^{-5}	16	0.96×10^{-5}	"	"	"	568.1×10^{-5}	6	0.32×10^{-5}	"	"	"	162.5×10^{-5}
n-Hexane	5	3	$<0.01 \times 10^{-5}$	"	"	"	$<5.9 \times 10^{-5}$	3	$<0.01 \times 10^{-5}$	"	"	"	$<5.9 \times 10^{-5}$	10	0.09×10^{-5}	"	"	"	45.7×10^{-5}
Methanol	5	2	$<0.05 \times 10^{-5}$	"	"	"	$<30 \times 10^{-5}$	16	3.65×10^{-5}	"	"	"	2160.2×10^{-5}	6	0.65×10^{-5}	"	"	"	330.2×10^{-5}
Methyl acetate	58		$<0.1 \times 10^{-5}$	"	"	"	$<59.2 \times 10^{-5}$		$<0.1 \times 10^{-5}$	"	"	"	$<59.1 \times 10^{-5}$		$<0.1 \times 10^{-5}$	"	"	"	$<50.8 \times 10^{-5}$
n-Pentane	58	2	$<0.01 \times 10^{-5}$	"	"	"	$<5.9 \times 10^{-5}$		$<0.01 \times 10^{-5}$	"	"	"	$<5.9 \times 10^{-5}$	34	0.23×10^{-5}	"	"	"	116.8×10^{-5}
	5	7	0.6×10^{-5}	"	"	"													
2-Propanol	5	2	$<0.05 \times 10^{-5}$	"	"	"	$<30 \times 10^{-5}$	16	1.05×10^{-5}	"	"	"	621.4×10^{-5}	6	$<0.05 \times 10^{-5}$	"	"	"	$<25.4 \times 10^{-5}$
	58	6	$<0.01 \times 10^{-5}$	"	"	"		1	$<0.01 \times 10^{-5}$	"				2	$<0.01 \times 10^{-5}$	"	"	"	$<5.0 \times 10^{-5}$

TABLE XXIIb

CALCULATION OF QUANTITIES EVOLVED FROM TEFLON WIRE INSULATION, #7

Compound	1 g_1	2 mg_1	3 mg/g	1 g_2	2 mg_2	3 mg/g	1 g_3	2 mg_3	3 mg/g	Average
Acetaldehyde	10.1778	<0.00059	$<58 \times 10^{-6}$	13.3608	<0.00059	$<44 \times 10^{-6}$	10.3965	<0.00051	$<49 \times 10^{-6}$	$<58 \times 10^{-6}$
Acetone	"	0.00267	264×10^{-6}	"	0.00994	74×10^{-6}	"	0.01158	1114×10^{-6}	707×10^{-6}[a]
Benzene	"	<0.00012	$<12 \times 10^{-6}$	"	<0.00012	$<9 \times 10^{-6}$	"	<0.00010	$<10 \times 10^{-6}$	$<12 \times 10^{-6}$
Chloroform	"	0.00296	291×10^{-6}	"	0.00385	288×10^{-6}	"	0.01092	1050×10^{-6}	543×10^{-6}
Ethanol	"	0.00036	35×10^{-6}	"	0.00568	425×10^{-6}	"	0.00162	156×10^{-6}	205×10^{-6}[b]
n-Hexane	"	<0.00006	$<6 \times 10^{-6}$	"	<0.00006	$<4 \times 10^{-6}$	"	0.00046	44×10^{-6}	$<6 \times 10^{-6}$
Methanol	"	<0.00030	$<29 \times 10^{-6}$	"	0.02160	1617×10^{-6}	"	0.00330	317×10^{-6}	$<1617 \times 10^{-6}$[c]
Methyl acetate	"	<0.00059	$<58 \times 10^{-6}$	"	<0.00059	$<44 \times 10^{-6}$	"	<0.00051	$<49 \times 10^{-6}$	$<58 \times 10^{-6}$[d]
n-Pentane	"	<0.00006	$<6 \times 10^{-6}$	"	<0.00006	$<4 \times 10^{-6}$	"	0.00167	161×10^{-6}	$<161 \times 10^{-6}$
2-Propanol	"	<0.00030	$<29 \times 10^{-6}$	"	0.00621	465×10^{-6}	"	<0.00025	$<24 \times 10^{-6}$	$<465 \times 10^{-6}$[e]
								<0.00005	$<5 \times 10^{-6}$	

[a]Includes a small amount of methyl acetate and 2-octene.

[b]Includes a small amount of methanol and 2-propanol.

[c]Includes a small amount of ethanol and 2-propanol.

[d]Includes a small amount of acetone and 2-octene.

[e]Includes a small amount of ethanol and methanol.

TABLE XXIIIa

CALCULATION OF QUANTITIES EVOLVED FROM B-FIBER,#8

		Sample #1						Sample #2						Sample #3					
		1	2	3	4	5	6	1	2	3	4	5	6	1	2	3	4	5	6
Compound	Col. No.	Ph/4	$\frac{mg}{5cc}$ @ atm	Pf mm	$\frac{Pf}{760}$	Vol.	mg	$\frac{Ph}{4}$	$\frac{mg}{5cc}$ @ atm	Pf mm	$\frac{Pf}{760}$	Vol.	mg.	$\frac{Ph}{4}$	$\frac{mg}{5cc}$ @ atm	Pf mm	$\frac{Pf}{760}$	Vol.	mg
Acetone	58	10.4	1.3 x 10^{-5}	1018.0	1.339	2010	699.8 x 10^{-5}	9.6	1.2 x 10^{-5}	1067.1	1.504	2010	660 x 10^{-5}	10.0	1.45 x 10^{-5}	1018.0	1.339	2018	676 x 10^{-5}
Benzene	58	10.4	0.82 x 10^{-5}	"	"	"	441.4 x 10^{-5}	9.5	0.75x 10^{-5}	"	"	"	423.3x10^{-5}	10.0	0.80 x 10^{-5}	"	"	"	432.3x 10^{-5}
Diethyl ether	58	8.0	0.215	"	"	"	115.7	2.0	<0.01	"	"	"	< 5.64	2.0	<0.01	"	"	"	< 5.40
Ethanol	5	5.0	0.22 x 10^{-5}	"	"	"	118.4 x 10^{-5}	4.5	0.22x 10^{-5}	"	"	"	124.2x10^{-5}	4.5	0.26 x 10^{-5}	"	"	"	140.5x 10^{-5}
n-Heptane	4	4.7	0.28 x 10^{-5}	"	"	"	150.7 x 10^{-5}	2.9	0.25x 10^{-5}	"	"	"	141.1x10^{-5}	3.0	0.26 x 10^{-5}	"	"	"	140.5 x10^{-5}
Hexane	4	8.8	0.07 x 10^{-5}	"	"	"	37.7 x 10^{-5}	9.5	0.08x 10^{-5}	"	"	"	45.2x10^{-5}	<1.0	<0.01 x 10^{-5}	"	"	"	< 5.4 x10^{-5}
Methyl acetate	4	23.2	2.0 x 10^{-5}	"	"	"	1076 x 10^{-5}	20.0	1.98x 10^{-5}	"	"	"	1116 x10^{-5}	16.5	1.6 x 10^{-5}	"	"	"	865 x10^{-5}
Methanol	4	1.0	< 05 x 10^{-5}	"	"	"	< 26.9 x 10^{-5}	2.8	<.05	"	"	"	< 28.2x10^{-5}	2.0	<.05	"	"	"	< 27.0 x10^{-5}
Methyl ethyl ketone	4	2.5	0.15 x 10^{-5}	"	"	"	80.7 x 10^{-5}	3.0	0.18x 10^{-5}	"	"	"	98.8x10^{-5}	3.5	0.19 x 10^{-5}	"	"	"	102.7 x10^{-5}
Methylcyclo-pentane	58	8.0	<0.01 x 10^{-5}	"	"	" <	5.64x 10^{-5}	2.0	< .01 x10^{-5}	"	"	"	< 5.64x10^{-5}	2.0	<0.01 x 10^{-5}	"	"	"	< 6.64x10^{-5}
Pentane	58	1.0	<0.01 x 10^{-5}	"	"	" <	5.4 x 10^{-5}	0.8	<. 01 x10^{-5}	"	"	"	< 5.4 x10^{-5}	3.0	<.01 x 10^{-5}	"	"	"	< 5.4 x10^{-5}

TABLE XXIIIb

CALCULATION OF QUANTITIES EVOLVED FROM B-FIBER, #8

Compound	1 g_1	2 mg_1	3 mg/gm	1 g_2	2 mg_2	3 mg/g	1 g_3	2 mg_3	3 mg/g	Average
		$\times 10^{-6}$	$\times 10^{-6}$		$\times 10^{-6}$	$\times 10^{-6}$		$\times 10^{-6}$	$\times 10^{-6}$	$\times 10^{-6}$
Acetone	11.8653	6998	589.8	10.0785	6600	660.8	10.4397	6755	647	<632.5
Benzene	"	4414	372	"	4233	420	"	4323	414	<402
Diethyl ether	"	1157	97.5	"	<56.4	5.60	"	<54.0	5.17	<97
Ethanol	"	1184	99.8	"	1242	123.2	"	140.5	134.6	119.2
n-Heptane	"	1507	127.0	"	1411	140.0	"	1405	134.6	133.9
Hexane	"	377	31.8	"	452	44.9	"	<54	5.17	<27.3
Methanol	"	<269	<22.7	"	<282	28.0	"	<270	25.9	25.5
Methyl acetate	"	10,760	906.9	"	11,160	1107.3	"	8650	828.5	947.6
Methylcyclopentane	"	<56.4	<4.75	"	<56.4	<5.60	"	<56.4	<5.40	<5.6
Methylethyl ketone	"	807	68.0	"	98.8	98.0	"	1027	98.4	88.1
Pentane	"	<54	4.55	"	<54	5.36	"	<54.	5.17	<5.36

TABLE XXIVa

CALCULATION OF QUANTITIES EVOLVED FROM SPONGE INSULATION BONDED WITH RTV, #9

		Sample #1						Sample #2						Sample #3					
		1	2	3	4	5	6	1	2	3	4	5	6	1	2	3	4	5	6
Compound	Col. No.	Ph/4	$\frac{mg}{5cc}$ @ atm	Pfmm	$\frac{Pf}{760}$	Vol.	Total mg	Ph/4	$\frac{mg}{5cc}$ @ atm	Pfmm	$\frac{Pf}{760}$	Vol.	Total mg	Ph/4	$\frac{mg}{5cc}$ @ atm	Pfmm	$\frac{Pf}{760}$	Vol.	Total mg
Acetone	58	9	$<0.05 \times 10^{-5}$	1022	1.3447	2003	$<26.9 \times 10^{-5}$	-	$<0.05 \times 10^{-5}$	1022	1.3447	2018	$<27.1 \times 10^{-5}$	0.5	$<0.05 \times 10^{-5}$	964	1.2684	2010	$<25.4 \times 10^{-5}$
Benzene	58	-	$<0.02 \times 10^{-5}$	"	"	"	$<10.7 \times 10^{-5}$	-	$<0.02 \times 10^{-5}$	"	"	"	$<10.8 \times 10^{-5}$	-	$<0.02 \times 10^{-5}$	"	"	"	$<10.1 \times 10^{-5}$
Cyclopentane	58	-	$<0.01 \times 10^{-5}$	"	"	"	$<5.3 \times 10^{-5}$	-	$<0.01 \times 10^{-5}$	"	"	"	$<5.4 \times 10^{-5}$	1	$<0.01 \times 10^{-5}$	"	"	"	$<5.0 \times 10^{-5}$
Ethanol	5	4	0.2×10^{-5}	"	"	"	107.7×10^{-5}	4	0.2×10^{-5}	"	"	"	108.5×10^{-5}	11	0.63×10^{-5}	"	"	"	321.2×10^{-5}
n-Hexane	5	1	$<0.01 \times 10^{-5}$	"	"	"	$<5.3 \times 10^{-5}$	4	$<0.01 \times 10^{-5}$	"	"	"	$<5.4 \times 10^{-5}$	26	0.43×10^{-5}	"	"	"	219.2×10^{-5}
Methanol	5	-	$<0.05 \times 10^{-5}$	"	"	"	$<26.9 \times 10^{-5}$	-	$<0.05 \times 10^{-5}$	"	"	"	$<27.1 \times 10^{-5}$	-	$<0.05 \times 10^{-5}$	"	"	"	$<25.4 \times 10^{-5}$
Methyl ethyl ketone	5	-	$<0.1 \times 10^{-5}$	"	"	"	$<53.8 \times 10^{-5}$	-	$<0.1 \times 10^{-5}$	"	"	"	$<54.2 \times 10^{-5}$	-	$<0.1 \times 10^{-5}$	"	"	"	$<50.9 \times 10^{-5}$
n-Octane	58	-	$<0.05 \times 10^{-5}$	"	"	"	$<26.9 \times 10^{-5}$	3	0.07×10^{-5}	"	"	"	37.9×10^{-5}	1	$<0.05 \times 10^{-5}$	"	"	"	$<25.4 \times 10^{-5}$
n-Pentane	58	61	0.49×10^{-5}	"	"	"	263.9×10^{-5}	45	0.34×10^{-5}	"	"	"	184.5×10^{-5}	37	0.26×10^{-5}	"	"	"	132.5×10^{-5}
2-Propanol	5	6	$<0.05 \times 10^{-5}$	"	"	"	$<26.9 \times 10^{-5}$	4	$<0.05 \times 10^{-5}$	"	"	"	$<27.1 \times 10^{-5}$	12	0.6×10^{-5}	"	"	"	305.9×10^{-5}

TABLE XXIVb

CALCULATION OF QUANTITIES EVOLVED FROM SPONGE INSULATION BONDED WITH RTV, #9

Compound	1 g_1	2 mg_1	3 mg/g	1 g_2	2 mg_2	3 mg/g	1 g_3	2 mg_3	3 mg/g	Average
Acetone	5.2111	<0.00027	$<52 \times 10^{-6}$	4.9917	<0.00027	$<54 \times 10^{-6}$	4.9488	<0.00025	$<50 \times 10^{-6}$	$<54 \times 10^{-6}$
Benzene	"	<0.00011	$<21 \times 10^{-6}$	"	<0.00011	$<22 \times 10^{-6}$	"	<0.00010	$<20 \times 10^{-6}$	$<22 \times 10^{-6}$
Cyclopentane	"	<0.00005	$<10 \times 10^{-6}$	"	<0.00005	$<10 \times 10^{-6}$	"	<0.00005	$<10 \times 10^{-6}$	$<10 \times 10^{-6}$
Ethanol	"	0.00108	207×10^{-6}	"	0.00108	216×10^{-6}	"	0.00321	649×10^{-6}	357×10^{-6}
n-Hexane	"	<0.00005	$<10 \times 10^{-6}$	"	<0.00005	$<10 \times 10^{-6}$	"	0.00219 <0.00005	442×10^{-6} $<10 \times 10^{-6}$	$<10 \times 10^{-6}$ [a]
Methanol	"	<0.00027	$<52 \times 10^{-6}$	"	<0.00027	$<54 \times 10^{-6}$	"	<0.00025	$<50 \times 10^{-6}$	$<54 \times 10^{-6}$
Methyl ethyl ketone	"	<0.00054	$<104 \times 10^{-6}$	"	<0.00054	$<108 \times 10^{-6}$	"	<0.00051	$<103 \times 10^{-6}$	$<108 \times 10^{-6}$
n-Octane	"	<0.00027	$<52 \times 10^{-6}$	"	0.00038	76×10^{-6}	"	<0.00025	$<50 \times 10^{-6}$	$<76 \times 10^{-6}$
n-Pentane	"	0.00264	507×10^{-6}	"	0.00034	68×10^{-6}	"	0.00026	52×10^{-6}	209×10^{-6}
2-Propane	"	<0.00027	$<52 \times 10^{-6}$	"	<0.00027	$<54 \times 10^{-6}$	"	0.00306	618×10^{-6}	$<618 \times 10^{-6}$

[a] Includes a very small amount of cyclopentane.

TABLE XXVa

CALCULATION OF QUANTITIES EVOLVED FROM SOLDER LUG INSULATION, #10

		Sample #1						Sample #2						Sample #3					
		1	2	3	4	5	6	1	2	3	4	5	6	1	2	3	4	5	6
Compound	Col. No.	PH/4	mg/5cc at atm	Pfmm	Pfmm/760	Vol.	Total mg	PH/4	mg/5cc at atm	Pfmm	Pf/760	Vol.	Total mg	PH/4	mg/5cc at atm	Pfmm	Pf/760	Vol.	Total mg
Acetaldehyde	5	13	$<0.1 \times 10^{-5}$	961	1.2644	2003	$<50.6 \times 10^{-5}$	7	$<0.1 \times 10^{-5}$	1012	1.3315	2010	$<53.5 \times 10^{-5}$	18	$<0.1 \times 10^{-5}$	961	1.2644	2010	$<50.8 \times 10^{-5}$
Acetone	58		$<0.05 \times 10^{-5}$	"	"	"	$<25.3 \times 10^{-5}$		$<0.05 \times 10^{-5}$	"	"	"	$<26.7 \times 10^{-5}$		$<0.05 \times 10^{-5}$	"	"	"	$<25.4 \times 10^{-5}$
	5		1.0×10^{-5}	"	"	"													
Benzene	58		$<0.02 \times 10^{-5}$	"	"	"	$<10.1 \times 10^{-5}$		$<0.02 \times 10^{-5}$	"	"	"	$<10.7 \times 10^{-5}$		$<0.02 \times 10^{-5}$	"	"	"	$<10.1 \times 10^{-5}$
n-Butane	58	4x48	1.72×10^{-5}	"	"	"	871.1×10^{-5}	4x43	1.53×10^{-5}	"	"	"	818.8×10^{-5}	4x85	3.0×10^{-5}	"	"	"	1524.8×10^{-5}
Chloroform	4	5	1.55×10^{-5}	"	"	"	785.0×10^{-5}	6	1.85×10^{-5}	"	"	"	990.1×10^{-5}	8	2.50×10^{-5}	"	"	"	1270.7×10^{-5}
Ethanol	5	2	0.06×10^{-5}	"	"	"	30.3×10^{-5}	3	0.12×10^{-5}	"	"	"	64.2×10^{-5}	3	0.12×10^{-5}	"	"	"	60.9×10^{-5}
n-Heptane	4	34	1.27×10^{-5}	"	"	"	643.2×10^{-5}	24	0.92×10^{-5}	"	"	"	492.3×10^{-5}	49	1.80×10^{-5}	"	"	"	914.9×10^{-5}
	58	32	0.48×10^{-5}	"	"	"	243.1×10^{-5}	24	0.33×10^{-5}	"	"	"	176.6×10^{-5}	44	0.7×10^{-5}	"	"	"	355.7×10^{-5}
Methanol	5	9.5	1.65×10^{-5}	"	"	"	835.7×10^{-5}	10	1.80×10^{-5}	"	"	"	963.3×10^{-5}	10	1.80×10^{-5}	"	"	"	914.9×10^{-5}
Methylcyclohexane	58	8	0.17×10^{-5}	"	"	"	86.1×10^{-5}	4	0.05×10^{-5}	"	"	"	26.7×10^{-5}	9	0.2×10^{-5}	"	"	"	101.6×10^{-5}
n-Octane	58	63.5	1.09×10^{-5}	"	"	"	552.0×10^{-5}	49	0.84×10^{-5}	"	"	"	449.5×10^{-5}	88	1.49×10^{-5}	"	"	"	757.3×10^{-5}
Toluene	5	16	1.42×10^{-5}	"	"	"	719.2×10^{-5}	14	1.28×10^{-5}	"	"	"	685.0×10^{-5}	35	2.9×10^{-5}	"	"	"	1474.0×10^{-5}
1,1,1-Trichloroethane	58	2	0.02×10^{-5}	"	"	"	10.1×10^{-5}		$<0.02 \times 10^{-5}$	"	"	"	$<10.7 \times 10^{-5}$	1	$<0.02 \times 10^{-5}$	"	"	"	$<10.1 \times 10^{-5}$

TABLE XXVb

CALCULATION OF QUANTITIES EVOLVED FROM SOLDER LUG INSULATION, #10

Compound	1 g_1	2 mg_1	3 mg/g	1 g_2	2 mg_2	3 mg/g	1 g_3	2 mg_3	3 mg/g	Average
Acetaldehyde	3.8461	<0.00051	$<133 \times 10^{-6}$	3.9786	<0.00054	$<136 \times 10^{-6}$	5.2938	<0.00051	$<96 \times 10^{-6}$	$<138 \times 10^{-6}$[a]
Acetone	"	<0.00025	$<65 \times 10^{-6}$	"	<0.00027	$<68 \times 10^{-6}$	"	<0.00025	$<47 \times 10^{-6}$	$<68 \times 10^{-6}$
Benzene	"	<0.00010	$<26 \times 10^{-6}$	"	<0.00011	$<28 \times 10^{-6}$	"	<0.00010	$<19 \times 10^{-6}$	$<28 \times 10^{-6}$
n-Butane	"	0.00871	2265×10^{-6}	"	0.00819	2058×10^{-6}	"	0.01525	288×10^{-6}	2401×10^{-6}
Chloroform	"	0.00785	2041×10^{-6}	"	0.00990	2488×10^{-6}	"	0.01271	2401×10^{-6}	2310×10^{-6}
Ethanol	"	0.00030	78×10^{-6}	"	0.00064	161×10^{-6}	"	0.00061	115×10^{-6}	118×10^{-6}
n-Heptane	"	0.00643 0.00243	1672×10^{-6} 632×10^{-6}	"	0.00492 0.00177	1247×10^{-6} 445×10^{-6}	"	0.00915 0.00356	1728×10^{-6} 672×10^{-6}	1549×10^{-6} 583×10^{-6}
Methanol	"	0.00836	2174×10^{-6}	"	0.00963	2420×10^{-6}	"	0.00915	1728×10^{-6}	2107×10^{-6}[b]
Methylcyclohexane	"	0.00086	224×10^{-6}	"	0.00027	68×10^{-6}	"	0.00102	193×10^{-6}	162×10^{-6}
n-Octane	"	0.00552	1435×10^{-6}	"	0.00450	1131×10^{-6}	"	0.00757	1430×10^{-6}	1332×10^{-6}[c]
Toluene	"	0.00719	1869×10^{-6}	"	0.00685	1722×10^{-6}	"	0.01474	2784×10^{-6}	2125×10^{-6}
1,1,1-Trichloroethane	"	0.00010	26×10^{-6}	"	<0.00011	$<28 \times 10^{-6}$	"	<0.00010	$<19 \times 10^{-6}$	$<28 \times 10^{-6}$

[a] *Includes significant amounts of n-octane and methylcyclohexane.*

[b] Peak includes a very small amount of benzene, which affects the average, less the spread.

[c] Peak includes 2-heptene and cyclohexane.

TABLE XXVI

Material	Item No.	Wt. before g.	Wt. after g.	Wt. change g.	Avg. change g.
Tygon 1, sample 1	1	20.5082	20.5411	+0.0329	
" 1, " 2		20.8489	20.8738	+0.0249	+.0304
" 1, " 3		20.0629	20.0972	+0.0343	
Tygon 2, sample 1	2	19.5965	19.6163	+0.0198	
" 2, " 2		18.9905	18.5145	+0.0240	+.0016
" 2, " 3		20.2524	20.2758	+0.0234	
Min-K 1301, sample 1	3	5.0459	5.0493	-0.0034	
" " 2		4.9199	4.9223	+0.0024	+.0016
" " 3		5.2928	5.2919	-0.0009	
Glass Cloth, sample 1	4	30.1853	30.1772	-0.0081	
" " 2		29.4242	27.4143	-0.0099	-.0064
" " 3		29.6989	29.6978	-0.0011	
Cohrlastic, sample 1	5	27.2296	27.2166	-0.0130	
" " 2		28.3902	28.3248	-0.0654	-.2334
" " 3		28.3615	28.9636	-0.6021	
Aluminum foil tape, 1	6	44.1224	44.1230	+0.0006	
" " " 2		50.2059	50.1938	-0.0121	-.0046
" " " 3		32.9041	32.9018	-0.0023	
Teflon Wire Mil-W-16878, Type E, 1	7	10.1778	10.1795	+0.0017	
" " 2		13.3608	13.3595	-0.0013	+.0007
" " 3		10.3965	10.3983	+0.0018	
Unbonded B Fiber, 1	8	10.0785	10.0769	-0.0014	
" " 2		11.8653	11.8603	-0.0050	-.0051
" " 3		10.4397	10.4307	-0.0090	
Bonded sponge insulation, 1	7	5.2111	5.2061	-0.0050	
" " 2 "		4.9917	4.9877	-0.0040	-.0039
" " 3		4.5659	4.5631	-0.0028	
Soldering lug insulation, 1	10	3.8461	3.8448	-0.0013	
" 2		3.9786	3.9796	+0.0010	+.0009
" 3		5.2938	5.2909	+0.0029	

8.0 SERIES B TESTS

8.1 DIFFERENTIAL THERMAL ANALYSES

The analyses reported were carried out on an American Instrument Company Differential Thermal Analyzer (DTA). The instrument produces a temperature difference between a sample and a reference (in this case air) during a period of fixed rate of heating giving a permanent record of the response of an X-Y recorder in graphical form. The instrument is capable of a maximum temperature of 1000°C at varying heating rates of 2, 4, 8 and 16°C per minute. Endotherms are shown as a negative change from the base line and exotherms as a positive change. On a heating cycle the usual recognized responses are moisture and solvent losses, phase changes, and some degradations as endotherms. Oxidations and some degradations occur as exotherms on a heating cycle. On a cooling cycle the phase changes occur as exotherms and most of the other responses do not occur. Some knowledge of the materials involved is necessary for interpretation of the results.

Response curves for the ten materials of construction are shown on the thermograms identified by sample number. In all cases the heating rates were arbitrarily designated as 16°C per minute. In samples, corrections were made for base line drift which is characteristic of the instrument and reference material. Two curves (Fig. 8 and 9) are shown for Sample No. 6, corrected and uncorrected. Sample No. 3 could not be corrected without providing a questionable result. Sample No. 8 would provide only minor change, if corrected.

Samples 1 and 2 represent plastic tubing and indicate melting ranges of material between 200°C and 300°C for No. 1 and between 280°C and 315°C for No. 2. In the range between 400°C and 430°C some loss of plasticizer or other material is indicated with continual degradation as the temperature is increased. It is likely that the material is completely charred near 350°C although no record was made until 1000°C was reached.

Sample No. 3, Min-K insulation, shows a broad, shallow exotherm between 450°C and 550°C which would imply a degradation of binding materials. The continued exotherm beyond 700°C could be attributed to degradation of the material.

Samples No. 4 and 5 represent types of silicone rubber and glass insulation. The endotherm peaking at about 500°C indicates melting without marked degradation. The exotherm at 650°C indicates degradations or oxidations of the silicone while the endotherm at 800°C suggests the melting of the glass substrate.

Sample No. 6 is represented by two curves, corrected and uncorrected. The latter curve more properly indicates volatilization and degradation of the adhesive on the aluminum foil starting at about 200°C with a maximum at 315°C. No marked change occurs until the melting point of aluminum (620°-625°C) is reached.

Sample No. 7 shows two endotherms at 320°C and 575°C. These would imply a change in crystallinity of the Teflon with degradative changes occurring between the endotherms. These are similar to DTA curves of Teflon reported in the literature.

Sample No. 8 ("B" fiber) is designated as unbonded fiber. The curve, however, implies a loss of some component as a broad shallow exotherm between 370°C and 540°C with a more complete degradation or oxidation above 600°C.

Sample No. 9 (Fig. 13) represents a double endotherm suggestive of fusion above 350°C or 550°C and degradation or oxidation above 650°C.

Sample No. 10 (Fig. 15) is designated as nylon sleeve insulation. The curve indicates a primary melting point at 300°C with a series of degradative changes at 350°C, 520°C and about 650°C. This curve suggests a mixture of materials rather than a single component. The curve is not remotely similar to standard curves of Nylon (Fig. 22 and 23). A representative of the company supplying this material indicated that it was probably polypropylene. Available DTA's of polypropylene (Fig. 24 and 25) do not match these obtained from sample Number 10 too well. Both have a marked endotherm at near 450°C so that it is consistent with DTA data that sample No. 10 has small amounts of polypropylene or that sample No. 10 could be composed largely of some of the new higher melting stereoregular polypropylenes. The endotherm at 450° is also typical of other polyolefins such as polyethylene.

Additional curves were included of the batting attached to the silicone coated fiberglas (No. 4) as the sample was available. These are duplicates showing apparent melting of the binder, but without degradation or volatilization to about 800°C. (Fig. 16 and 17).

Table XXVII summarizes the results of complete ignition of the samples to 1000°C. The DTA charts also show the weight gain or loss of sample and the appearance of the residue. If no weight is shown part of the substance spattered out of the cup and any weight differences would be meaningless. A weight loss indicates that at least this portion of the sample is lost when heated to the upper temperature limit. A weight gain indicates either that the substance was oxidized and the oxide was nonvolatile. No weight change indicates either that the substance has lost nothing, or less likely, that any loss has been exactly compensated for by a gain in weight due to oxidation.

TABLE XXVII

SUMMARY OF DTA DATA

ITEM[a]	WEIGHT	WEIGHT CHANGE	% CHANGE	
1 Tubing	-	-	-	Burned leaving puffy black ash
2 Tubing	-	-	-	Burned leaving puffy black ash
3 Min-K	-	-	-	Fused
4 Red cloth	.1749	-.0555	-31.7%	Burned leaving black cloth
5 White cloth				Burned leaving black cloth
6 Aluminum tape	.7267	-.0368	-16.2%	Burned leaving black cinder
7 Wire	.1984	-.1163	-58.6%	Burned leaving black residue
8 Batting	-	-	-	Fused
9 Sponge	-	-	-	Burned leaving black residue
10 Wire end	-	-	-	Burned leaving black residue
11(4b) Blue batting	.0696	+.0005	+0.71%	Fused

[a] Identification of items in Table II

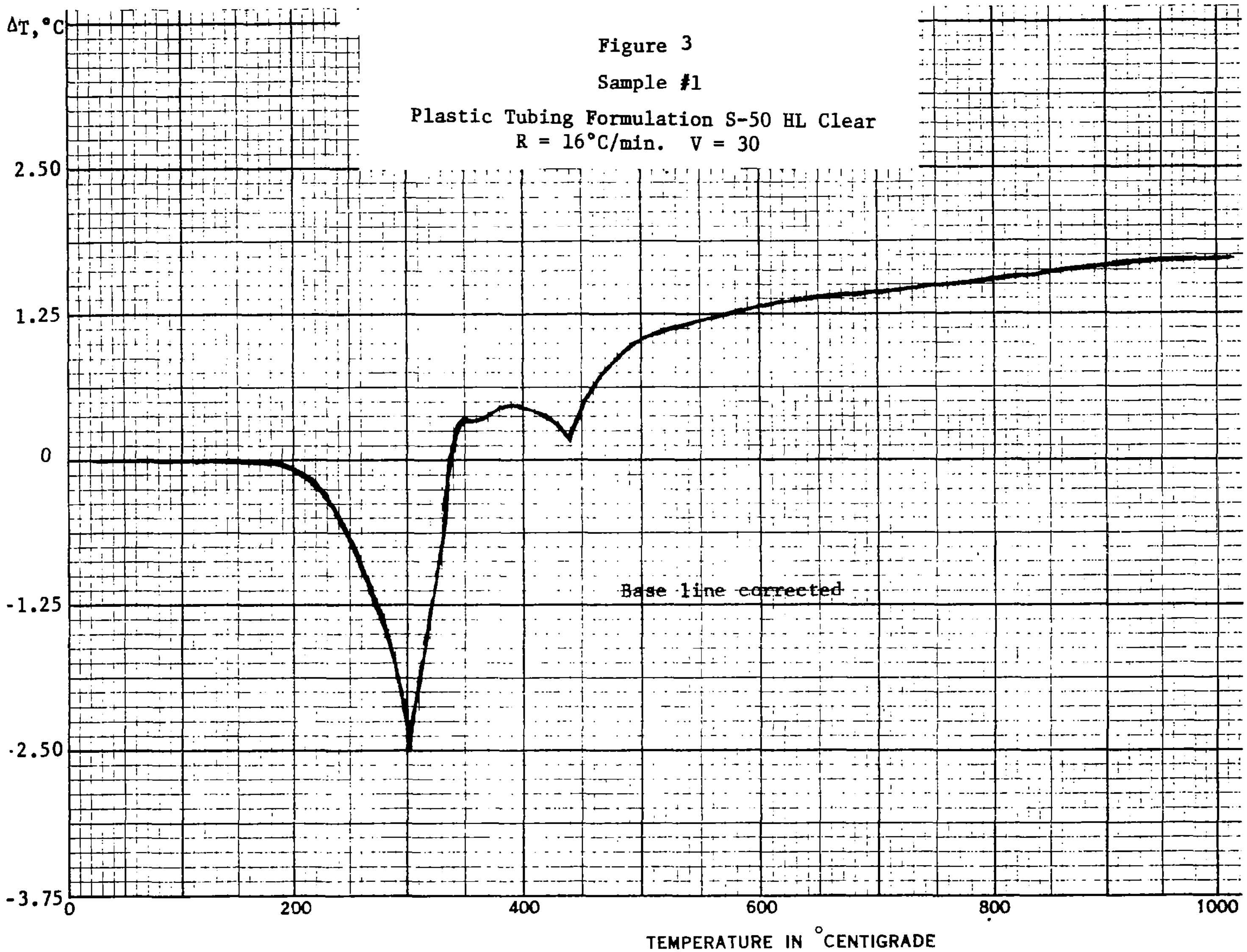


Figure 3
Sample #1
Plastic Tubing Formulation S-50 HL Clear
R = 16°C/min. V = 30

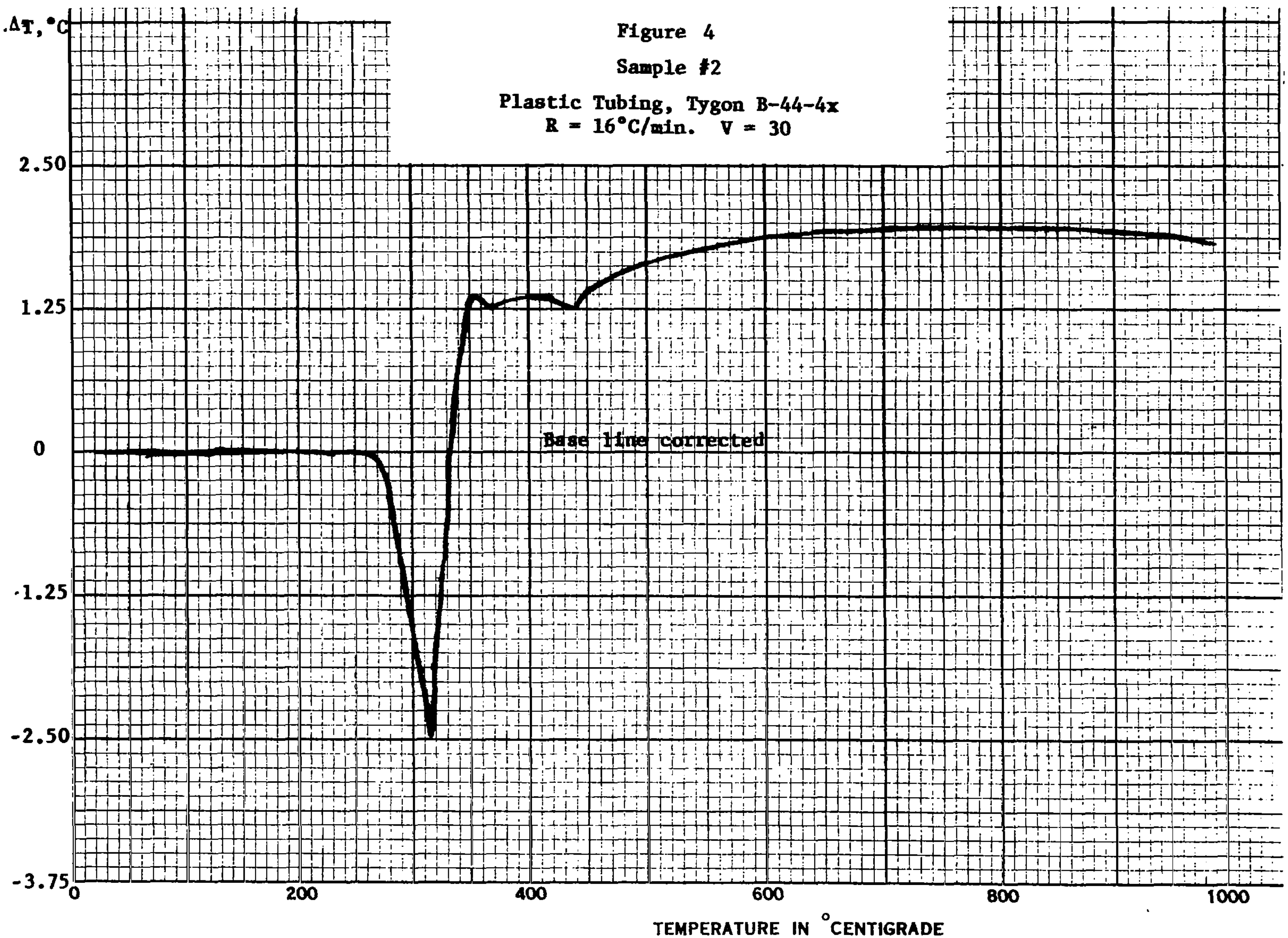


Figure 4

Sample #2

Plastic Tubing, Tygon B-44-4x
R = 16°C/min. V = 30

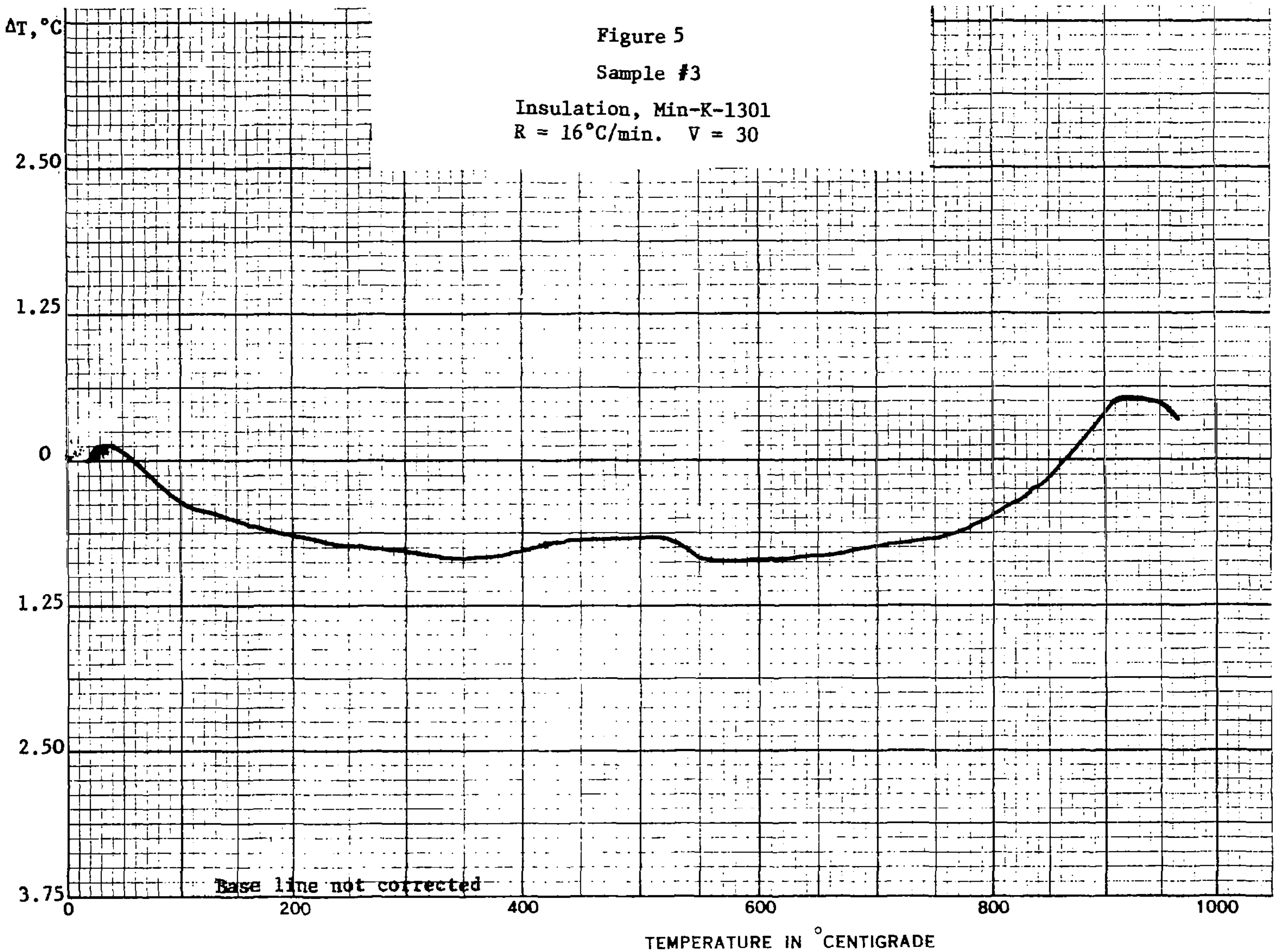


Figure 5

Sample #3

Insulation, Min-K-1301
R = 16°C/min. V = 30

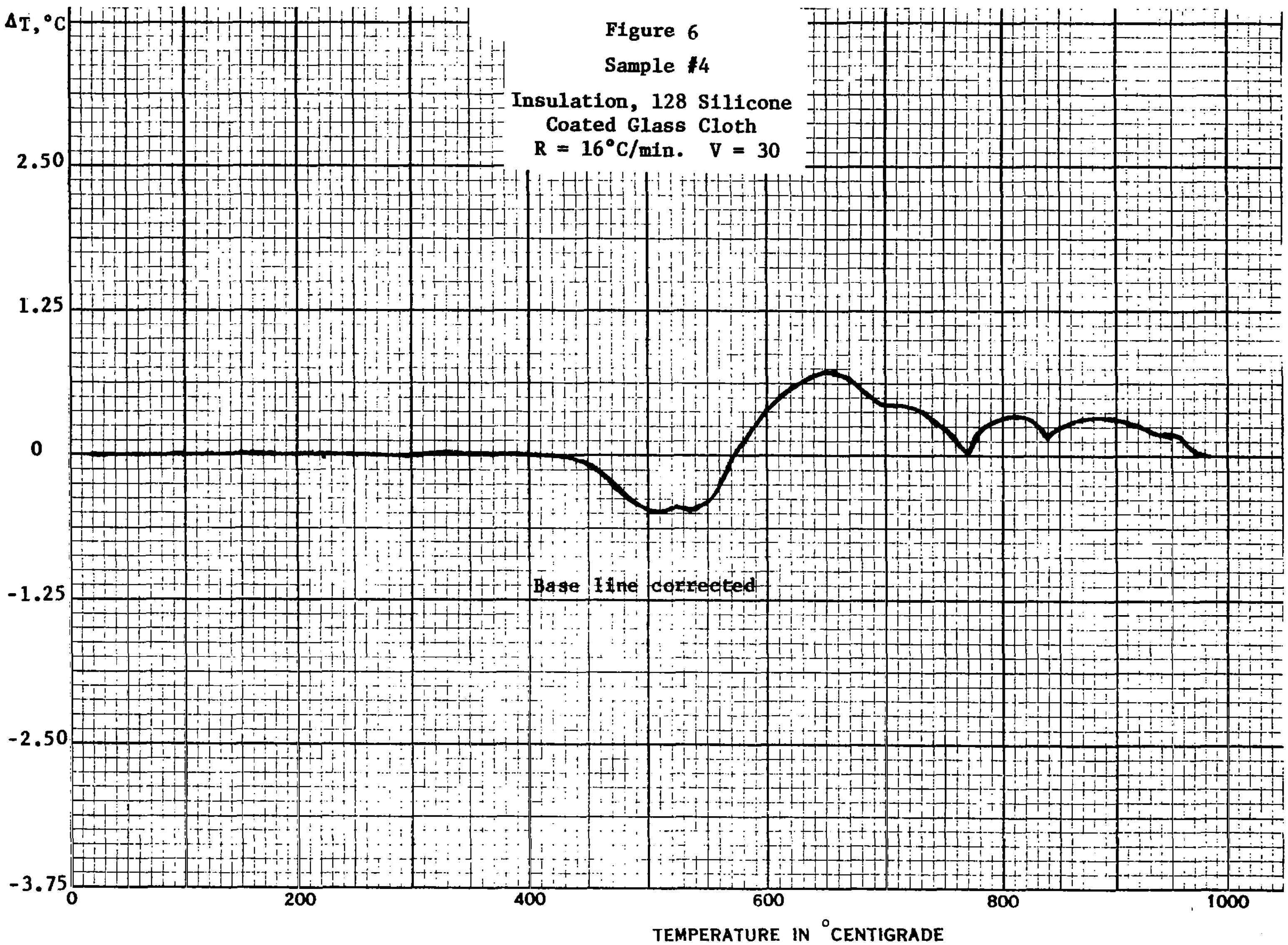


Figure 6
Sample #4
Insulation, 128 Silicone Coated Glass Cloth
R = 16°C/min. V = 30

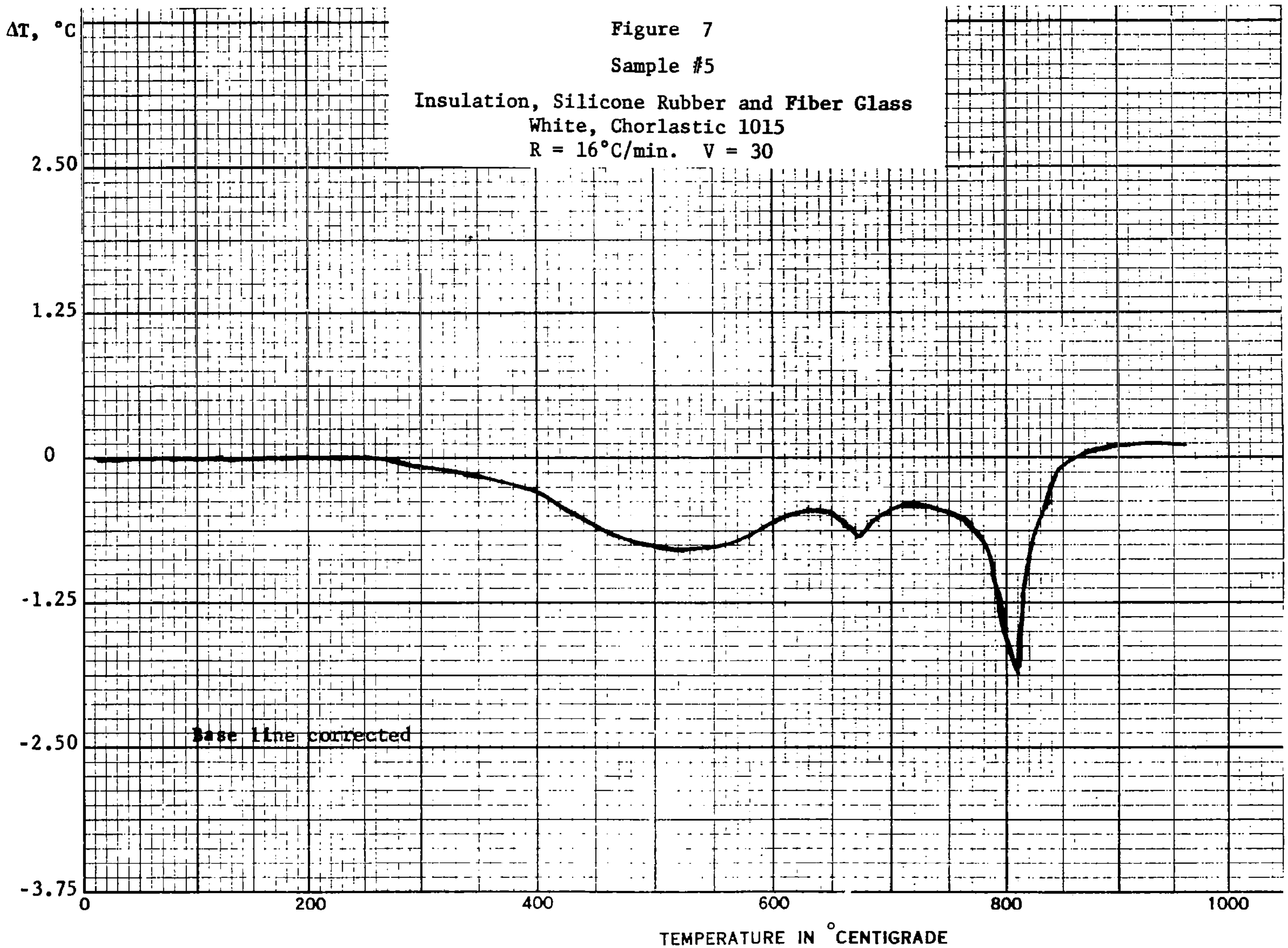


Figure 7

Sample #5

Insulation, Silicone Rubber and Fiber Glass
White, Chorlastic 1015
R = 16°C/min. V = 30

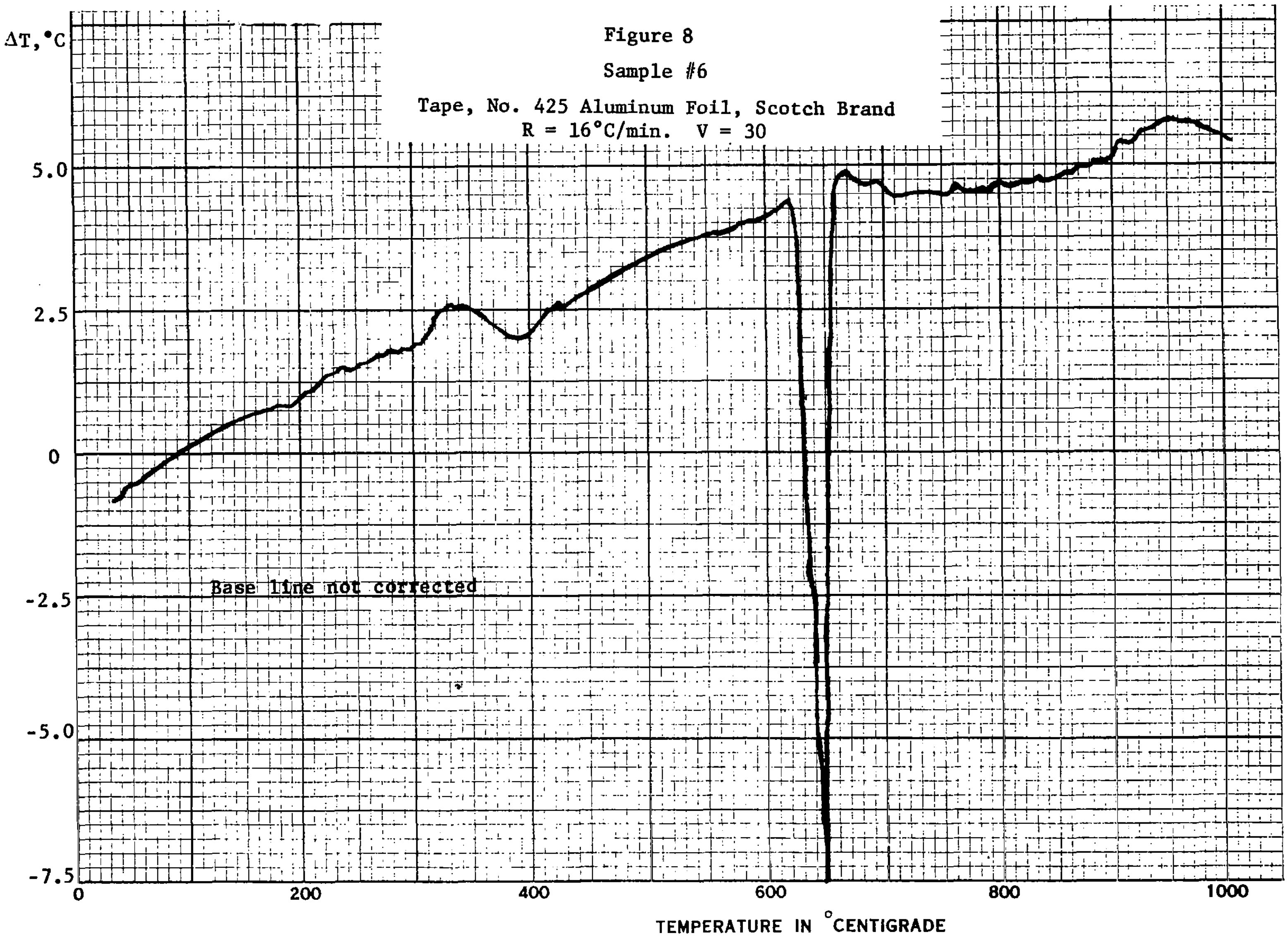


Figure 8
Sample #6
Tape, No. 425 Aluminum Foil, Scotch Brand
R = 16°C/min. V = 30
ΔT, °C
5.0
2.5
0
-2.5
-5.0
-7.5
0
200
400
600
800
1000
Base line not corrected
TEMPERATURE IN °CENTIGRADE

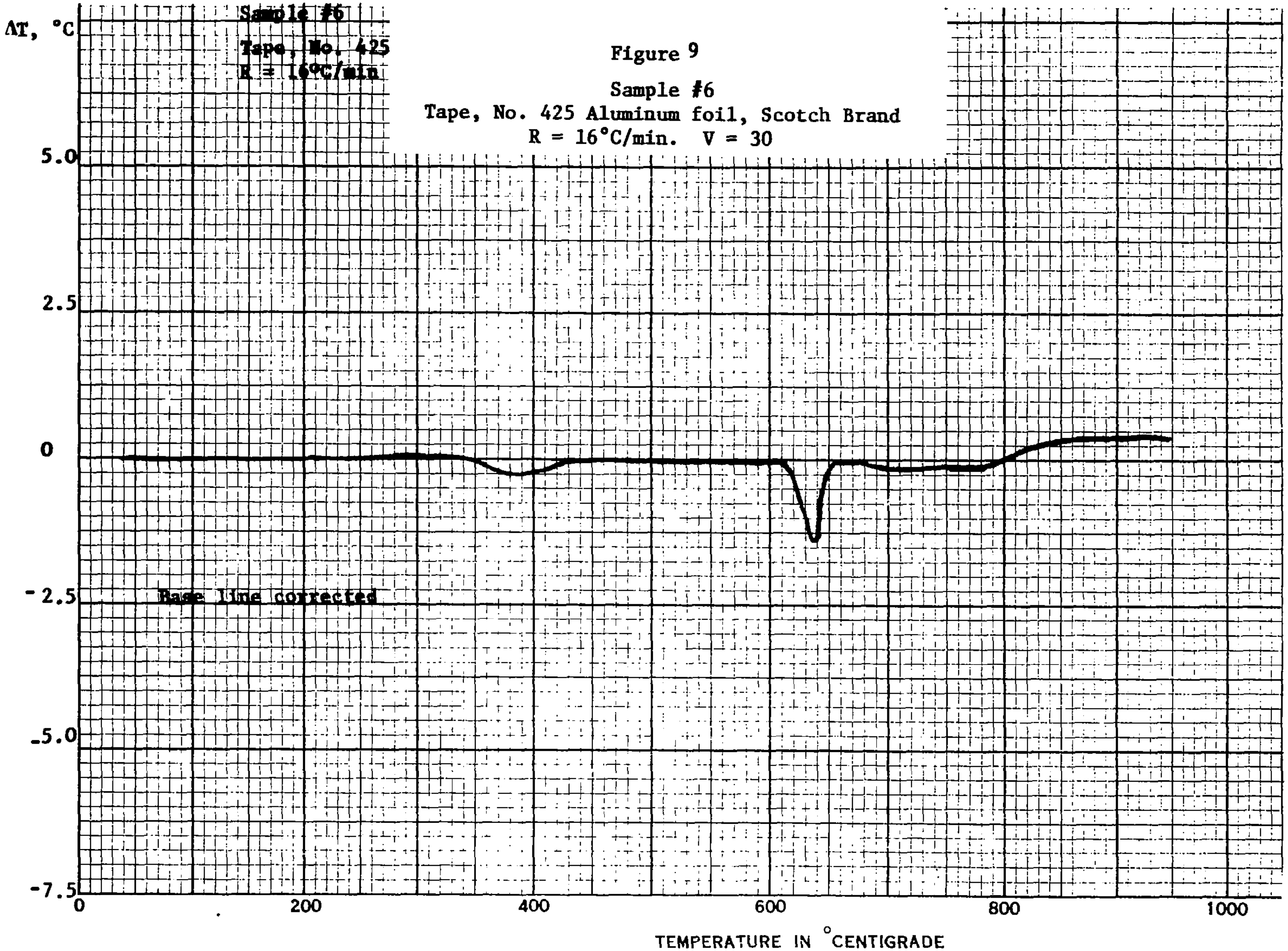


Figure 9

Sample #6

Tape, No. 425 Aluminum foil, Scotch Brand

R = 16°C/min. V = 30

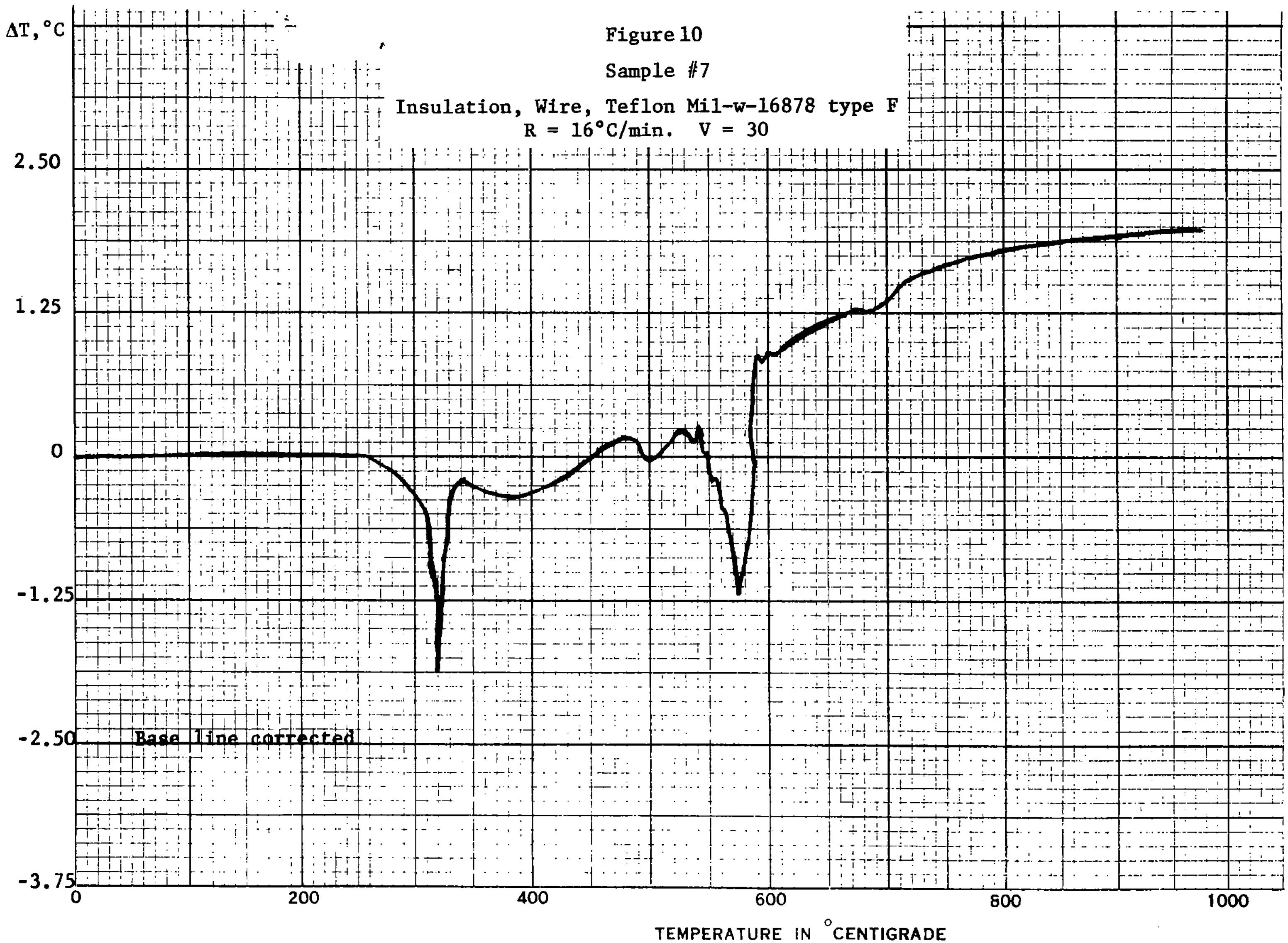


Figure 10

Sample #7

Insulation, Wire, Teflon Mil-w-16878 type F
R = 16°C/min. V = 30

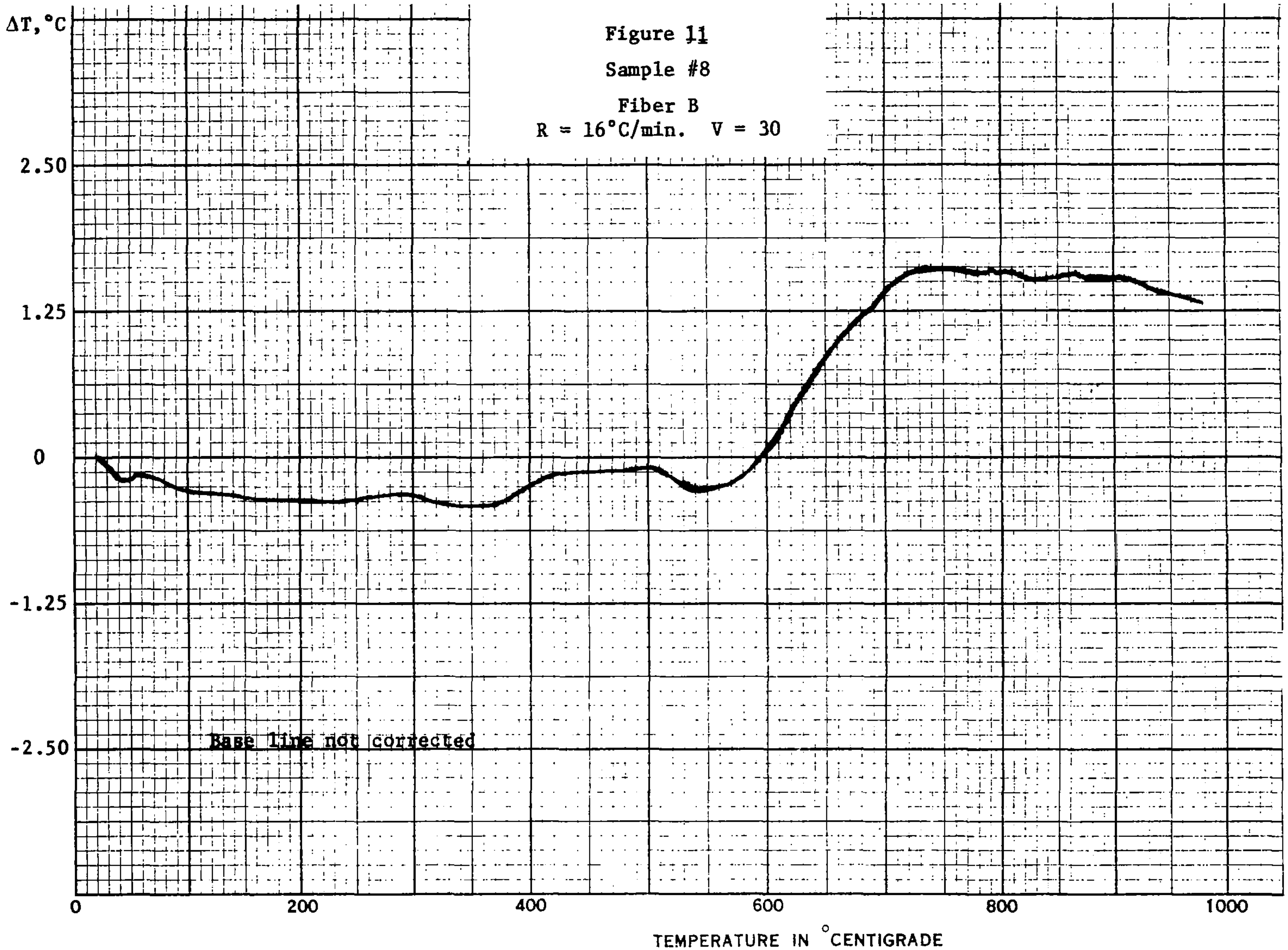


Figure 11
Sample #8
Fiber B
R = 16°C/min. V = 30
ΔT, °C
2.50
1.25
0
-1.25
-2.50
Base line not corrected
0
200
400
600
800
1000
TEMPERATURE IN °CENTIGRADE

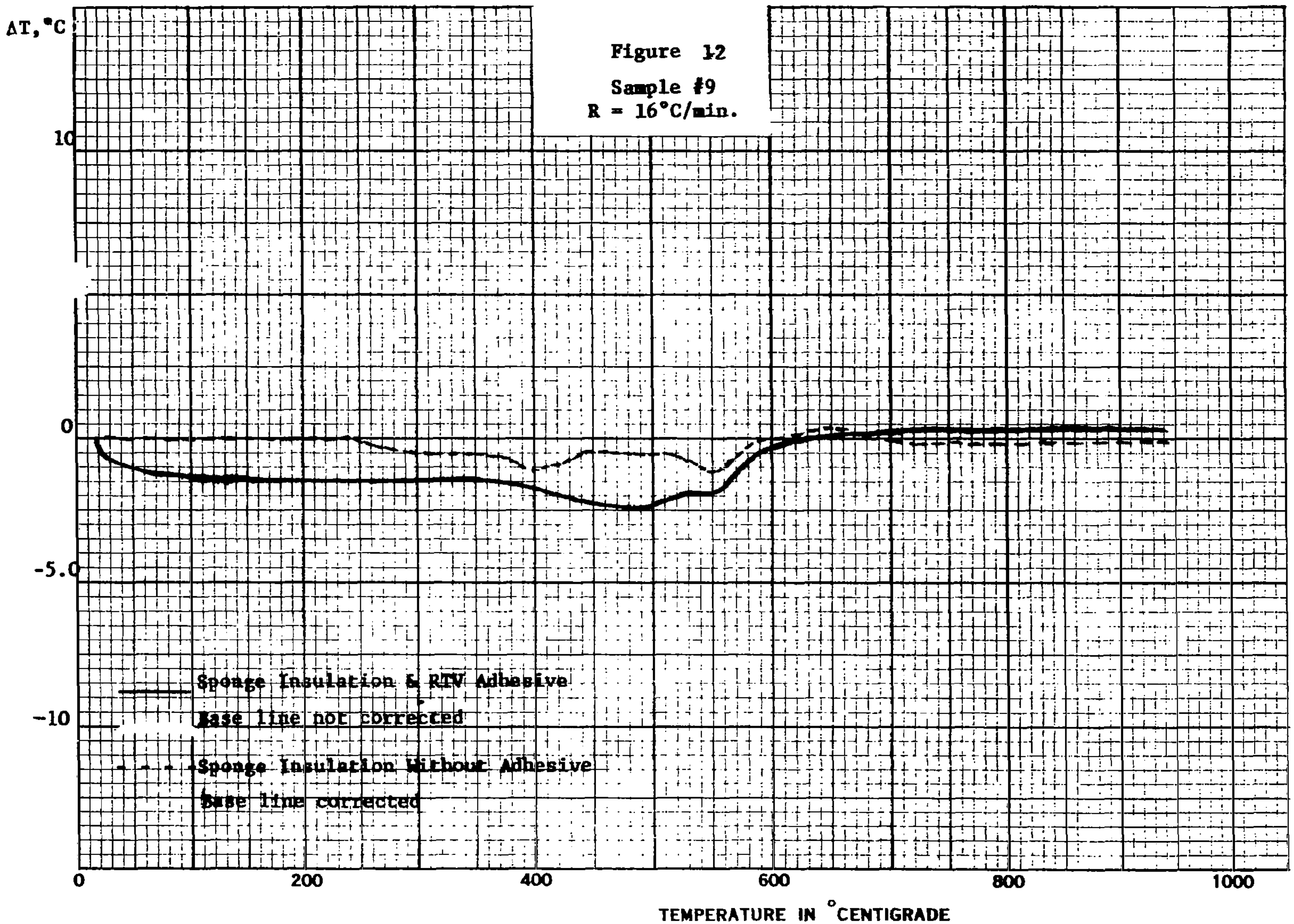
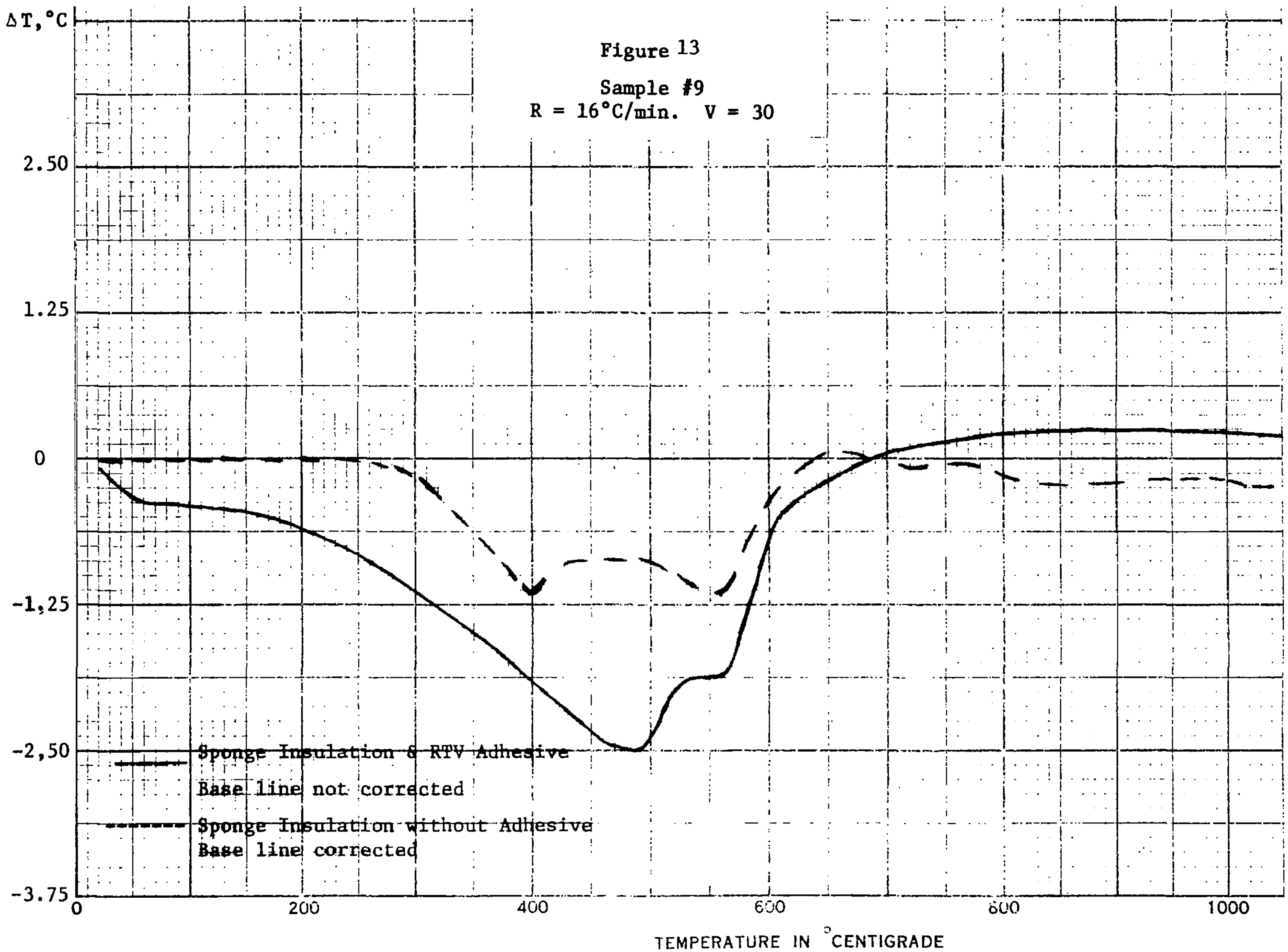


Figure 12
Sample #9
R = 16°C/min.


ΔT,°C
Figure 13
Sample #9
R = 16°C/min. V = 30
2.50
1.25
0
-1,25
-2,50
-3.75
Sponge Insulation & RTV Adhesive
Base line not corrected
Sponge Insulation without Adhesive
Base line corrected
0
200
400
600
800
1000
TEMPERATURE IN °CENTIGRADE

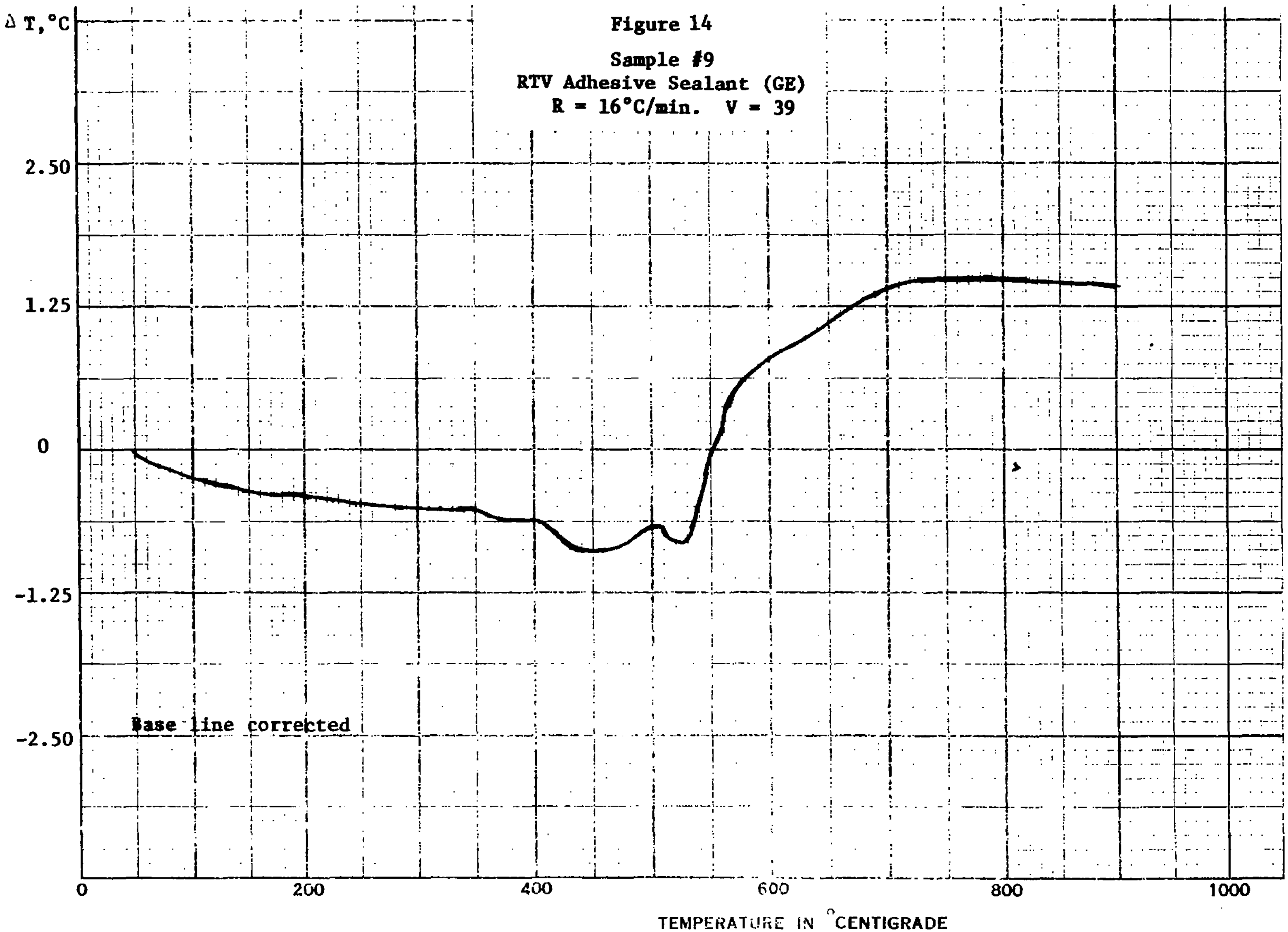


Figure 14

Sample #9
RTV Adhesive Sealant (GE)
R = 16°C/min. V = 39

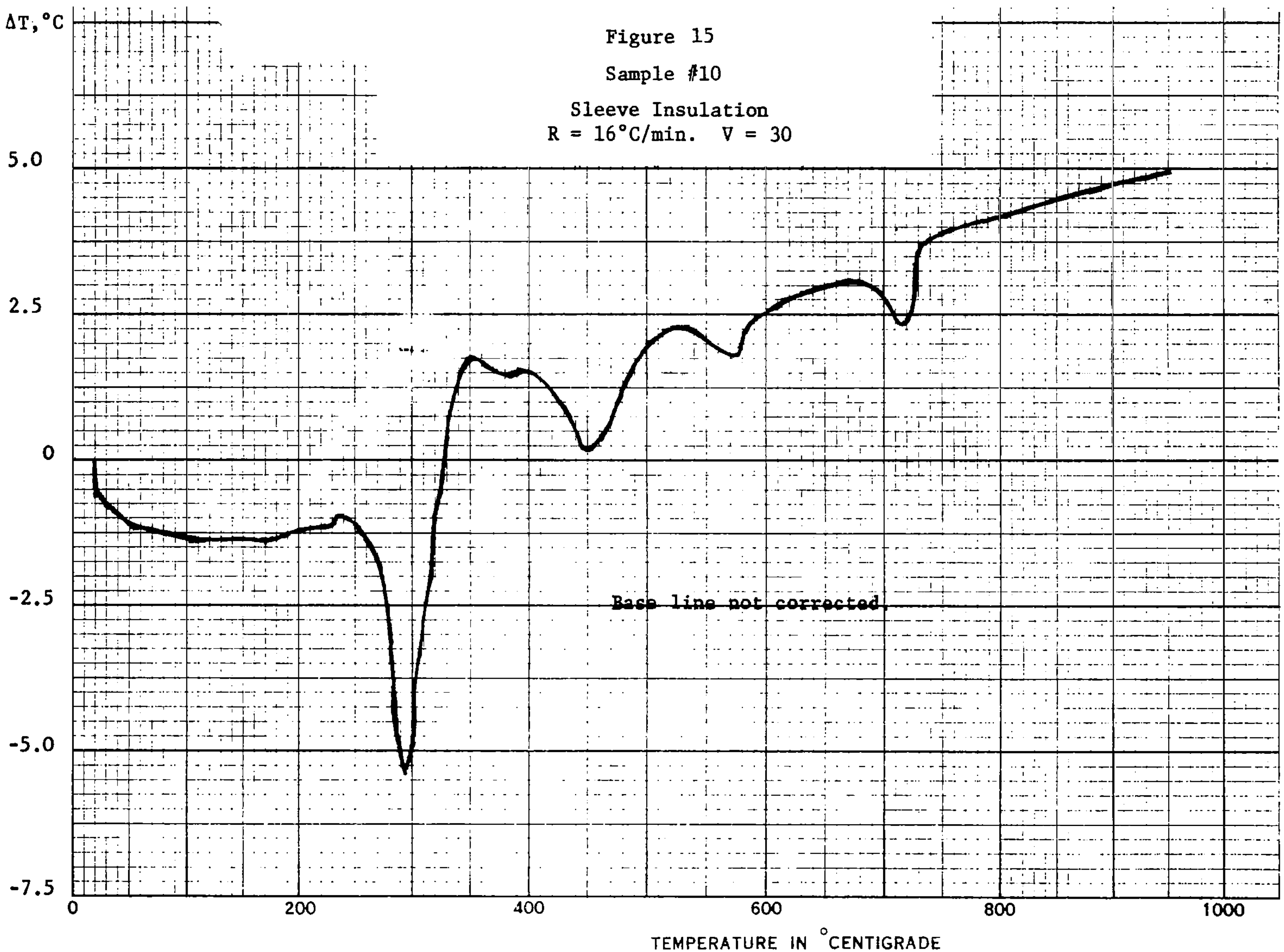


Figure 15

Sample #10

Sleeve Insulation

R = 16°C/min. V = 30

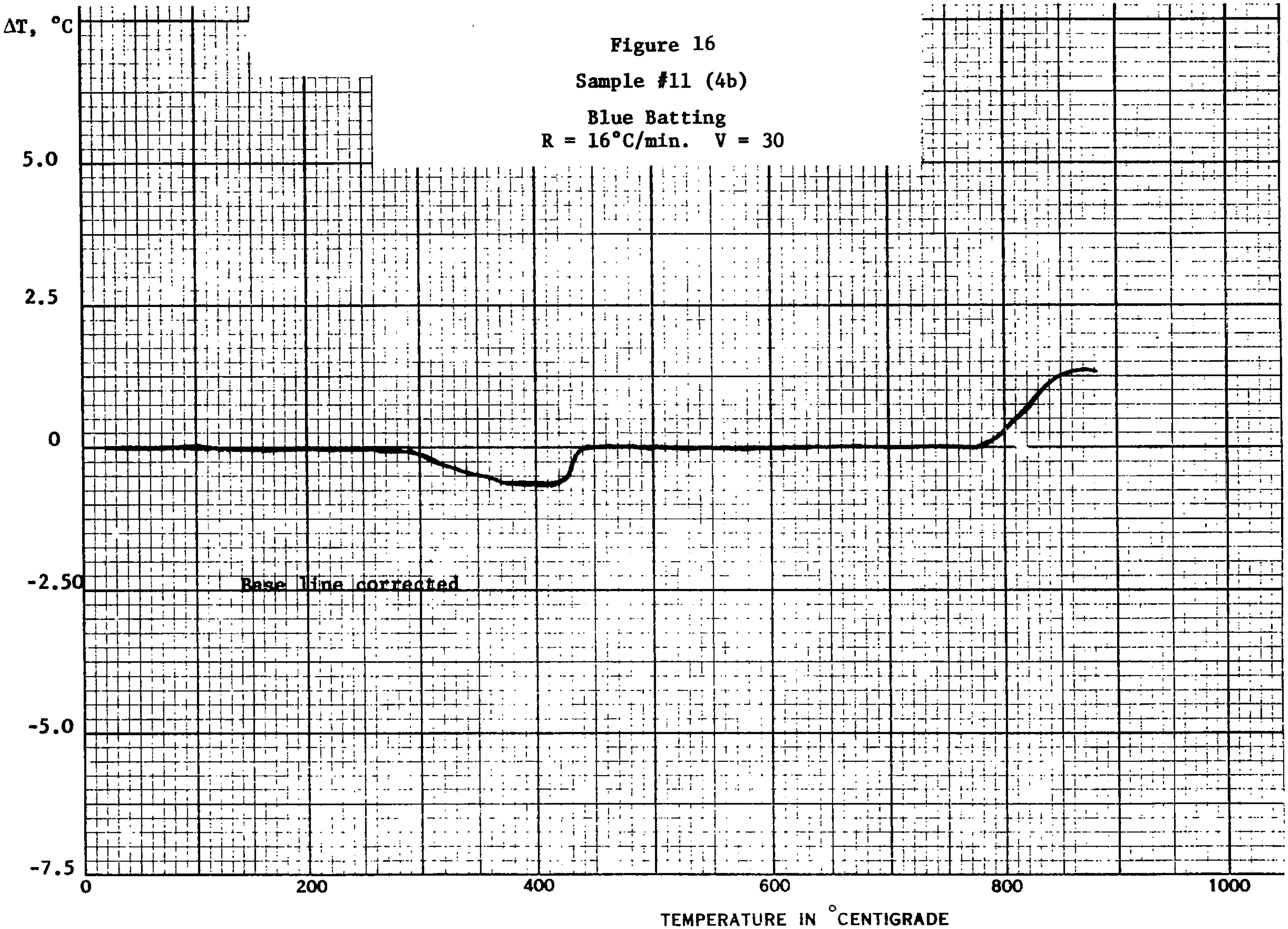


Figure 16

Sample #11 (4b)

Blue Batting

R = 16°C/min. V = 30

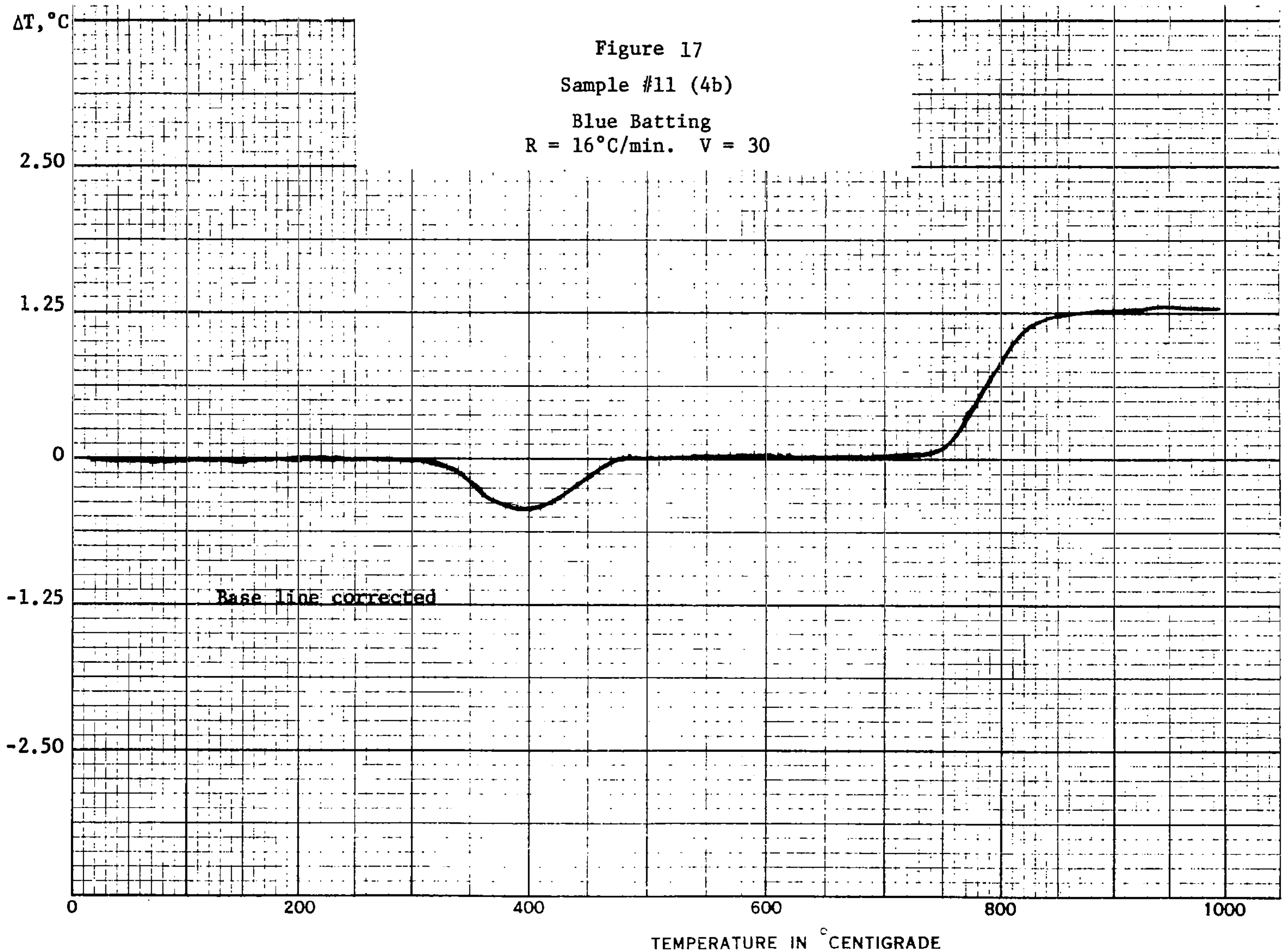


Figure 17
Sample #11 (4b)
Blue Batting
R = 16°C/min. V = 30

8.2 DIFFERENTIAL SCANNING CALORIMETRIC ANALYSIS

Three of the samples of materials of construction were analyzed using a Perkin-Elmer Differential Scanning Calorimeter. (D.S.C.). Although the D.S.C. cannot be operated at as high a temperature as the Aminco DTA previously used, it has advantages in the examination of pyrolytic or other changes at temperatures below 500° C. The instrument is designed such that the areas prescribed by the curves are directly proportional to the quantity of heat evolved or absorbed and may be calculated in terms of calories per gram of substance. Additionally, the instrument contains a thermal conductivity cell which allows detection of gas evolution and correlation of the gas evolution with exotherms or endotherms. The DSC also is more sensitive for the same amount of base line drift than the DTA. The samples were run on the DSC in an atmosphere of argon. The original DTA curves were carried out using the samples in air. For convenience in reading temperature in both degrees centigrade and degrees absolute (Kelvin) are plotted along the x-axis of the DSC.

In general the DSC data verifies the DTA data. The only significant difference is in sample No. 5 where the DSC shows very little if any endotherm and the DTA shows a significant endotherm. This can be rationalized by assuming that the endotherm shown in the DTA is an oxidative decomposition. Since the atmosphere is argon in the DSC, no oxidative decomposition occurs. Although there were no significant endotherms or exotherms in the DSC of sample 5 there was minor gas evolution. The gas has a heat capacity smaller than the heat capacity of argon.

The DSC and DTA data show that no apparent decompositions occur below 220° C (428° F). This indicates that there should be no <u>large</u> amounts of volatile products given off by these substances when heated to 200° F. Long periods of exposure could slightly mitigate this effect. Series "A" tests confirmed this conclusion for although several samples gave off a large number of compounds, the total amount of volatiles given off was small - both on an absolute basis and compared relative to the sample size.

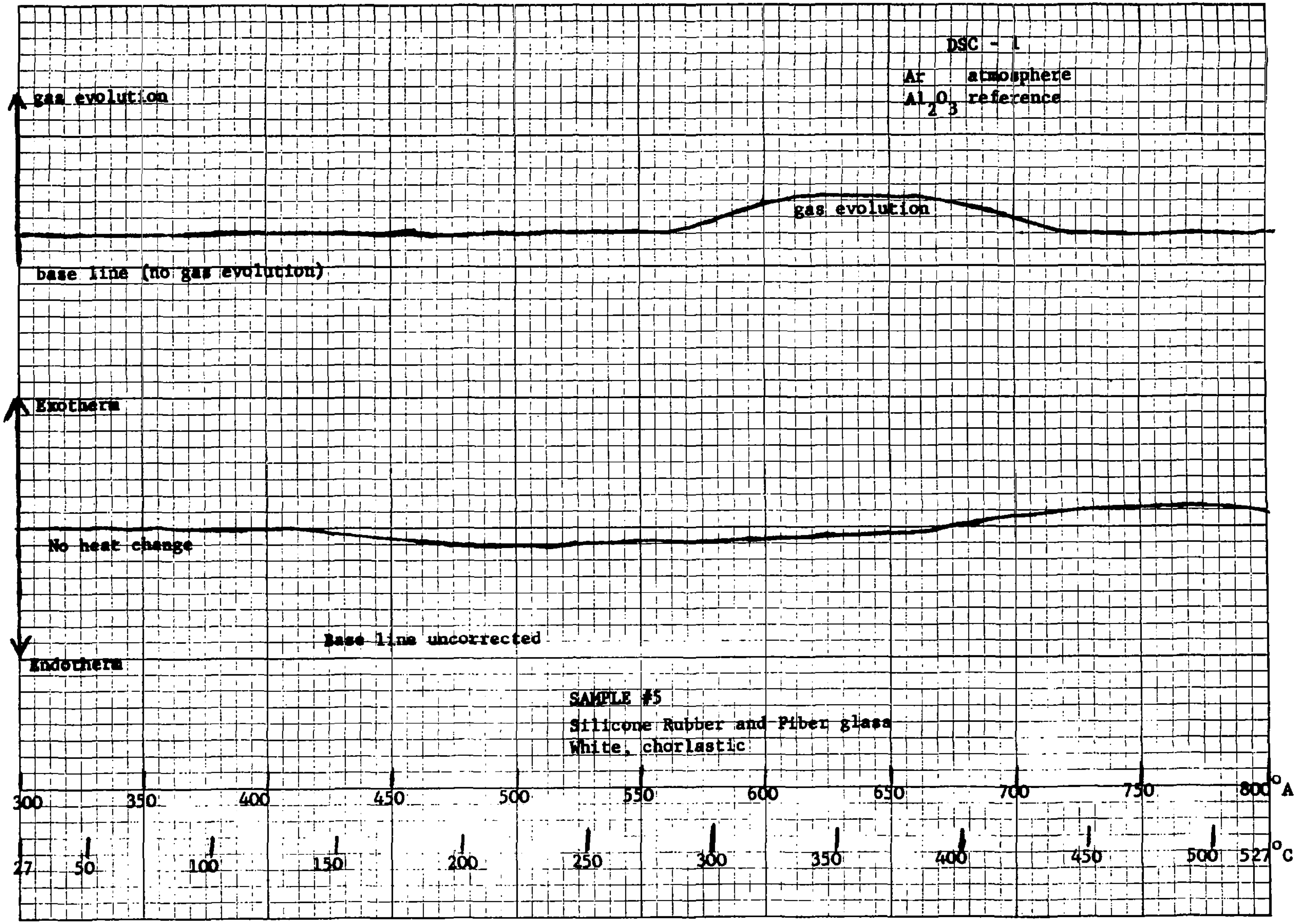

DSC - 1
Ar atmosphere
Al_2O_3 reference
gas evolution
gas evolution
base line (no gas evolution)
Exotherm
No heat change
Base line uncorrected
Endotherm
SAMPLE #5
Silicone Rubber and Fiber glass
White, chorlastic
300 350 400 450 500 550 600 650 700 750 800 °A
27 50 100 150 200 250 300 350 400 450 500 527 °C

Figure 18

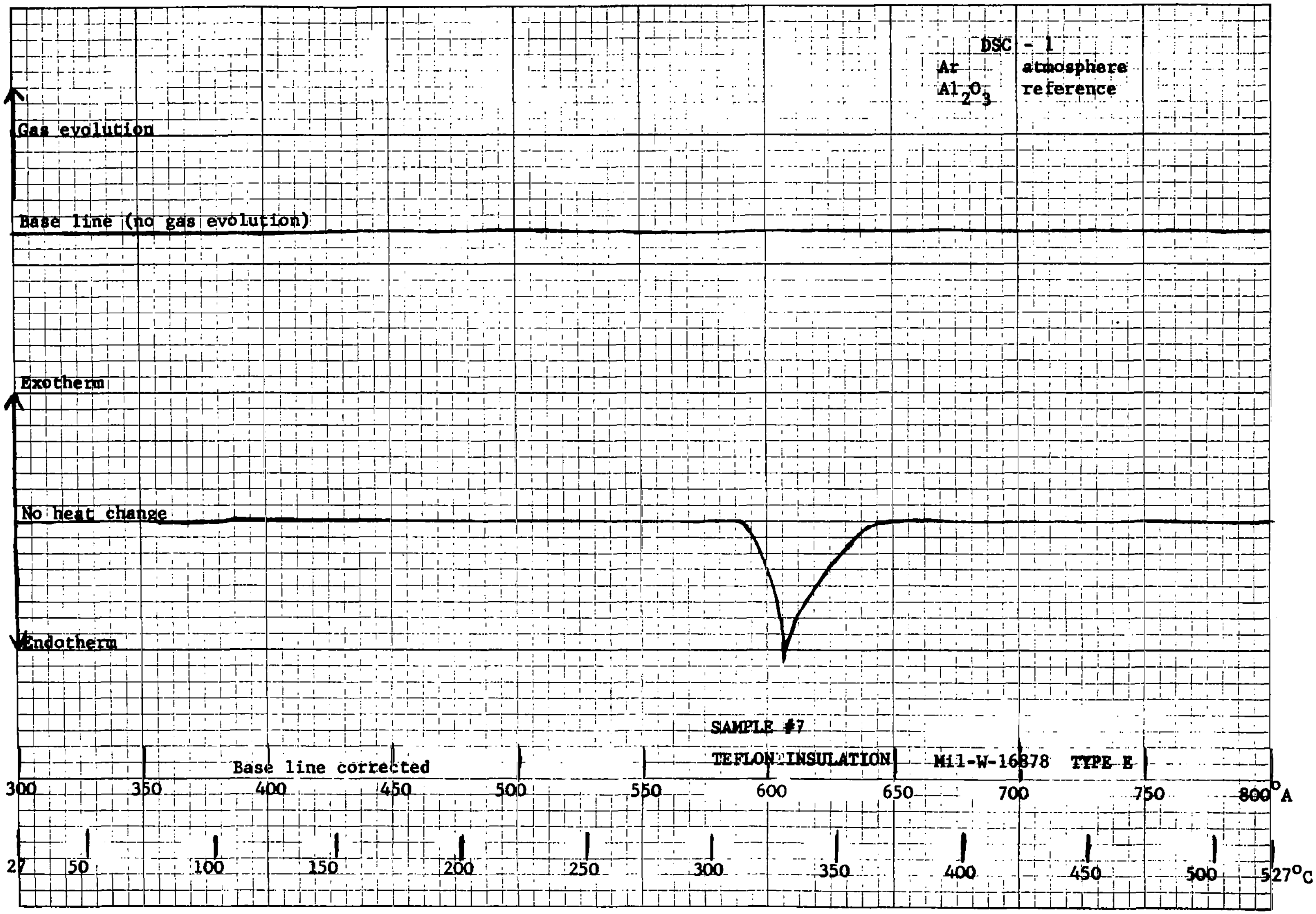

DSC - 1
Ar atmosphere
Al_2O_3 reference
Gas evolution
Base line (no gas evolution)
Exotherm
No heat change
Endotherm
SAMPLE #7
TEFLON INSULATION Mil-W-16878 TYPE E
Base line corrected
300 350 400 450 500 550 600 650 700 750 800 °A
27 50 100 150 200 250 300 350 400 450 500 527°C

Figure 19

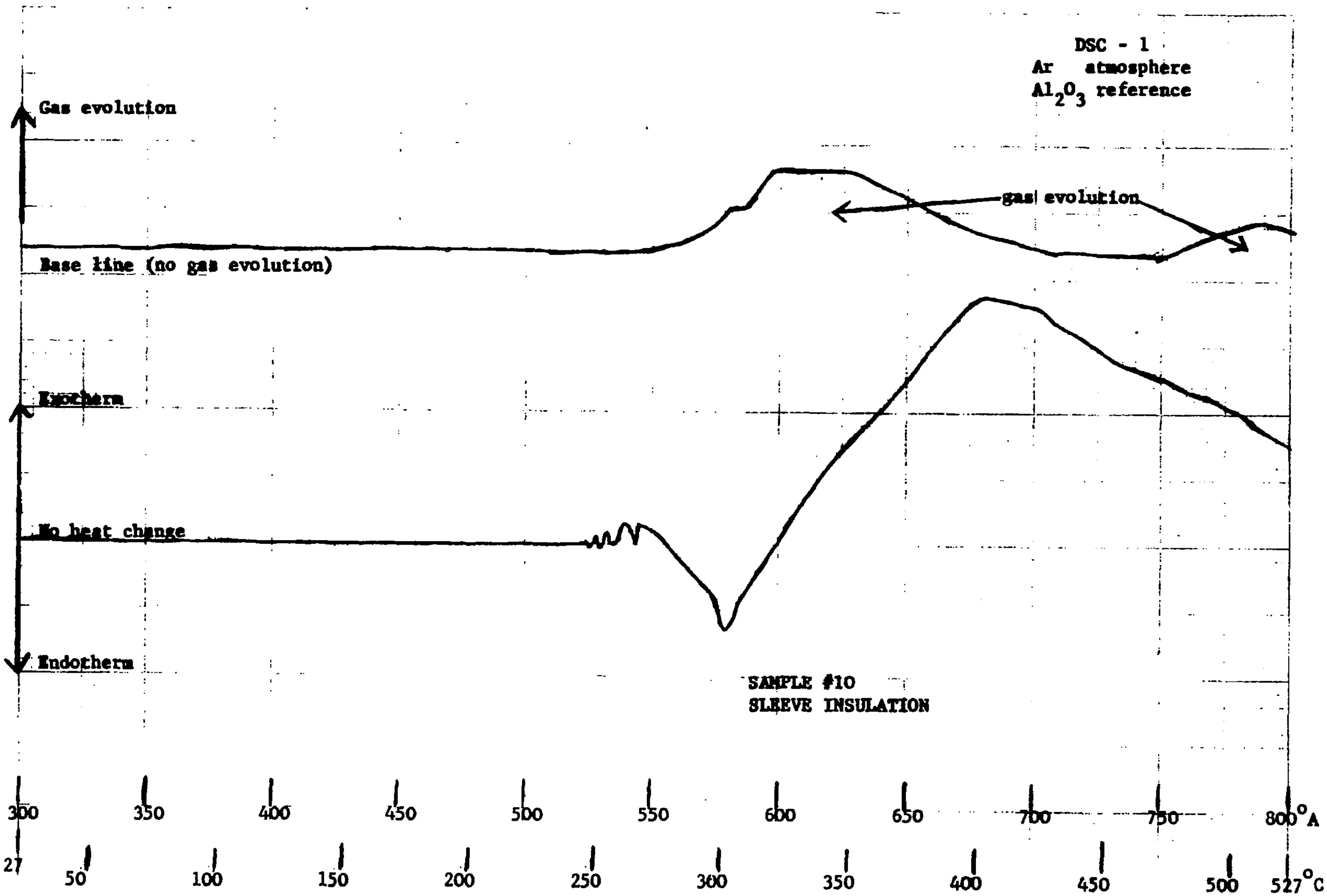

DSC - 1
Ar atmosphere
Al_2O_3 reference
Gas evolution
gas evolution
Base line (no gas evolution)
Exotherm
No heat change
Endotherm
SAMPLE #10
SLEEVE INSULATION
300
350
400
450
500
550
600
650
700
750
800°A
27
50
100
150
200
250
300
350
400
450
500
527°C

Figure 20

9.0 SERIES C TESTS

Eight of the samples showed evidence of more extensive degradation, decomposition, and/or oxidation in the series C tests. The data from the series C tests is given in Table XXVIII. The comparison of weight changes for the samples that showed significant difference in the two tests is given in Table XXIX. Besides weight change, the samples also gave evidence of decomposition by extensive charring (items 1,2, and 10), discoloration (item 6) and noticeable odor on opening the container (items 1,2,6,9 and 10). Except for a slight change in compliance of the two Tyon tubings, none of the samples of series A test showed such readily observable evidence of decomposition.

Only items 3 (Min-K fiber) and 7 (Teflon insulation) showed no evidence of change on the Series C Tests, and the Teflon was exposed in Series C at a temperature lower than the 200°F of the Series A tests. Since the Series C tests correspond more closely to the temperature the materials could be exposed to, it would seem advisable to repeat the analysis for gas-off products after exposure at temperatures comparable to those used in the Series C Tests.

TABLE XXVIII

SERIES C TESTS

Material	Temperature	Item No.	Wt. before g.	Wt. after g.	Wt. change	Comments: Appearance	Comments: Vapors
Tygon #1	350°F	1	17.9967	14.9159	-3.0808	Black	Odor of H_2S, HCl, and others
Tygon #2	350°F	2	13.9381	12.4570	-1.4811	Black	Odor of H_2S, HCl, and others
Min-K-1301	1200°F	3	5.0544	5,0521	-0.0023	No noticeable change	No noticeable odor
Silicone coated glass cloth	350°F	4	30.3134	30.2578	-0.0556	No change	No noticeable odor
Cohrlastic	350°F	5	30.0462	30.0338	-0.0124	No noticeable change	No noticeable odor
Aluminum foil tape	440°F	6	50.4516	50.0727	-0.3789	Adhesive side of tape turned brown	Slight burned odor
Teflon covered wire	150°	7	10.4844	10.4846	+0.0002	No noticeable change	No noticeable odor
B-Fiber	600°F	8	9.8411	9.8166	-0.0245	No noticeable change	No noticeable odor
Sponge insulation bonded with R.T.V.	350°F	9	5.4893	5.4661	-0.0232	No noticeable change	Slight acid odor.
Insulation, solder lug	350°F	10	5.1411	3.0705	-2.0706	Turned black	Strong odor of H_2S and HCl

TABLE XXXIX

COMPOUNDS THAT SHOWED SIGNIFICANT DIFFERENCES IN THE WEIGHT CHANGES BETWEEN SERIES C AND SERIES A TESTS

Material	Item No.	Average weight change of 3 exposures in Series A Tests	Weight change Series C Tests	Weight difference of material losses of two series of tests - (Column 4 - Column 3)
Tygon	1	+0.0304	-3.0808	2.9472
Tygon	2	+0.0224	-1.4811	1.5035
Silicone coated glass cloth	4	-0.0064	-0.0556	0.0492
Cohrlastic	5	-0.0392 -0.2334 [a]	-0.0124 -0.0124	-0.0268 -0.2210
Aluminum foil tape	6	-0.0046	-0.3789	+0.3743
B-Fiber	8	-0.0051	-0.0245	+0.0194
Bonded sponge insulation	9	-0.0039	-0.0232	+0.0183
Solder lug insulation	10	+0.0009	-2.0706	+2.0715

[a] Average of two closest values.

10.0 DISCUSSION OF EXPOSURE RESULTS

10.1 BEHAVIOR OF CONSTRUCTION MATERIALS

The materials found from the exposure test probably have three main sources: (1) from outgassing of solvents remaining from solvent treatment or cleaning of the materials before use or fabrication, or from solvents used in fabrication of the material; (2) from outgassing or decomposition of plasticizers or other additives; (3) from chemical changes of the structural materials.

The alkanes and halohydrocarbons undoubtedly are due to source (1). The ketones, alcohols, aldehydes, and esters could also be from this source. The ketones, alcohols and aldehydes (especially acrolein) could also be from decomposition or oxidation of any of the materials. The alkenes are probably produced by decomposition of esters:

$$R_2CH\text{-}\underset{\substack{\backslash \\ O\text{-}\underset{\overset{\|}{O}}{C}\text{-}R''}}{CHR'} \xrightarrow{\Delta} R_2C = CHR' + R''CO_2H$$

where R,R',R" can be alkyl, aryl, or hydrogen decomposition of esters. The hexamethylcyclotrisiloxane could be a low molecular weight component of silicone oils used for siliconizing or it could be an additive. Siloxanes are sometimes used to help development of uniform pore size in the preparation of foams.

The greatest amount of material was given off by the Cohrlastic,#5. However, almost all this was ethanol which is relatively nontoxic. Other than ethanol Cohrlastic gave off less gaseous materials than any of the other substances examined except the silicone glass cloth, #4, and the sponge insulation bonded with RTV, #9. Other materials gave off large amounts of compounds. These materials listed in order of the amounts given off are: Tygon (#1), solder lug insulation, (#10), Tygon (#2), and Aluminum Foil Tape #6.

Information was obtained from the manufacturer about fabrication and treatment of four of the materials.

Before extruding from a mold the Teflon is washed with naphtha. This would account for the presence of the methyl- and dimethylcyclohexanes, pentane, and hexane. Since the Teflon is extruded at a high temperature the 2-octene and benzene could come from cracking of some of the hydrocarbons in the naphtha. The small traces of other solvents probably came from solvents used to clean the wire prior to covering with Teflon. This, however, is speculation as no information on treatment of the wire was obtained from the manufacturer.

The aluminum tape had an adhesive made from 2-ethylhexyl acrylate (and other monomers whose identity is proprietary). The adhesive was applied to the tape with a solvent mixture of benzene, toluene, heptane, methyl ethyl ketone, and possible acetone. The hexane and possibly all the other alkanes found were probably contaminants in the heptane used in the solvent mixture. The 2-ethyl-1-hexene was undoubtedly from decomposition of the 2-ethylhexyl acrylate. The other alkenes may have come from similar decompositions of other esters used in the formulation. The manufacturer could not (or would not) offer any explanation of the two compounds found in highest concentration, carbon tetrachloride and 2-propanol. These may have been used to clean the aluminum tape prior to application of the adhesive.

The unbonded B-Fiber is claimed to be pure asbestos. The final treatment before shipment is with water. The other compounds may have come from other solvents used before the water treatment. Some of them were present in such small amounts that they could have come from adsorption of gaseous materials from the environment.

The only information obtained about the Min-K fiber was that it is pure asbestos bonded with up to 6% Bakelite. Because of the very small weight change in the Class C test, it is doubtful that this much Bakelite was used in the sample examined. The formaldehyde found was probably from the Bakelite.

10.2 OCCURRENCE OF COMPOUNDS

Methyl ethyl ketone, benzene, acetaldehyde, and acetone were found in large amounts from the Tygon #1. Carbon tetrachloride was found in large amounts from Tygons 1 and 2, and aluminum foil tape. Alkanes were found in large amounts from Tygons 1 and 2, and Solder Lug insulation. Methanol was found in large amounts from Teflon wire and Solder Lug insulations. Ethanol was found in large amounts from Tygon 1 and Cohrlastic. Chloroform and trichloroethane were found in large amounts from the Solder Lug Insulation.

11.0 CHAMBER ATMOSPHERE SAMPLES

11.1 GENERAL PROCEDURE

The same gas chromatographic instruments, columns, and procedures described in Sections 7.1 and 7.2 were used for the analysis of the atmospheric samples. Mass and infrared spectroscopy were not used because the samples were too dilute and too small. The quantitative data and retention times used for the analysis are the same as those used from the analysis of the atmospheres of the exposure experiments (Series A Tests).

The mixtures were analyzed quantitatively by comparing peak heights of the grab samples with peak heights obtained from known quantities of the same compounds on one of the columns used for qualitative analysis (Tables XXXIV and XXXV).

Tables XXX-XXXIII indicate the compounds identified and the method used to identify them. A compound was not considered identified until its retention times were found to be the same as the retention times of a known standard on at least two different stationary phases. In addition, if a compound is indicated as definitely or probably being present, none of the gas chromatographic data could indicate that it was not present. For example, if there was a peak with the same retention time as a known standard on as many as five different stationary phases, and the unknown had no peak on a sixth stationary phase where a compound of its concentration should have a peak and there was no possibility of masking by other strong peaks or by the background, then it was concluded that the unknown was not the compound in question. When this occurred, a new search was begun for a compound consistent with both the mass spectral and the gas chromatographic data. In many cases, no more confirmations of the unknown could be obtained on the liquid phases attempted because of low concentration and/or because of masking by components of higher concentration or by high background. For such non-confirmations an NC is written in Tables III-XII. This is not to be considered a negative result. As indicated above, if a negative result was found for a proposed compound, the compound was not entered in the tables and a new search was begun for the unknown responsible for that peak.

A compound is recorded as definitely being present (D) if the minimum requirements (outlined above and in the work statement) were met plus at least one more retention time coincident with that of the known standard found. If only the minimum requirements were met, the compound was recorded as probably being present (P). If less than the minimum requirements were met, but evidence indicated that the compound might be present, the contaminant was recorded as possibly being present (M). For most of the compounds identified, there were at least three retention times identical with a known standard.

Tables XXX to XXXIII summarize the qualitative results of the four cryogenically trapped samples. The number 18 refers to samples that were trapped on July 18, before the air purifier was turned on and the number 19 refers to samples trapped July 19 after the air purifier was turned on. The Dry Ice trap is indicated by CO_2; the liquid Nitrogen trap by LN_2.

The concentrations are those found relative to acetone in the liquid nitrogen trap on the first day since this was the highest concentration of any compound found in the cryogenic traps. The acetone in this trap is assigned a value of 1000 and all the other concentrations are given relative to it. Because of problems in obtaining quantitative transfer from the cryogenic traps, there may be some error in the relative amounts and they should be interpreted to indicate orders of magnitude although the results may be more accurate than this. Since compounds in the same class adsorb similarly to stainless steel and fiber glass surfaces, the relative concentrations for compounds in the same class are probably fairly accurate. For more accurate estimation of the various quantities of contaminants, the figures obtained from the grab samples (Table XXXIV) should be used. However, since this was an unconcentrated sample, several of the minor components could not be detected and an estimation of their concentration can be obtained only from the cryogenically trapped samples.

The large amount of acetone found in all four cryogenic traps and in the two grab samples was in such high concentration that most of it was undoubtedly an artifcat. A probable source of this contaminant was incomplete evacuation of the collection cylinders which had been cleaned with acetone.

Where the quantities were present in large enough concentrations to be accurately determined, or even reasonably estimated, the concentrations of most of the materials were found to be significantly less on the second day. This was shown by both the cryogenically trapped and grabbed samples. Note, however, that the early peaks, probably due to C_2-C_4 alkenes and alkanes, show a higher concentration of these materials on the second day. Some compounds found from the first day's exposure were not present in high enough concentration to be found from the second day's exposure.

TABLE XXX

DRY ICE TRAP 1ST DAY (7/18/66)

	Certainty of Identification	Approx. relative quan.	Retention				Times			
			Carbowax 5	Apiezon 4	Triscyano 58	QF1 61	QF1 62	DEGS 60	Alcon 56b	Silicone Grease 63
Acetone	D	70	5.15	1.9	13.1	2.6	2.53	8.27	3.8	2.00
Isopropanol	D	10	8.4	1.9	13.1			12.2	6.98	2.40
Ethanol	D	4	9.0	1.7	14.7			9.3	6.79	2.00
Methanol	D	23	NC	1.25?	NC			7.45		NC
Carbon Tetrachloride	P	1	NC	4.7	5.7				10.2	
Chloroform	P	< 1	9.9?	3.2	NC				5.82	
Cyclohexane	P	2	3.3	NC	2.2			NC	3.8	7.05
1,2-Dimethylcyclohexane	M		NC	4.7	4.68					
Ethylbenzene	M	< 0.1	18.0?	NC			7.5	NC	18.2	
2-Ethyl-1-hexene	M		3.9	NC					NC	
n-Heptane	P	5	3.0?	NC	1.5	2.6		NC	NC	8.4
Hexamethylcyclotrisiloxane	P	< 0.4	4.2	NC	5.7				6.8	13.5
Hexane	P	4	2.6	NC	1.05		NC	NC	NC	NC
2-Hexene	P	< 0.1	NC	3.2	1.4				3.5?	1.95
Methylcyclopentane	M	< 0.1	3.9	NC	1.95					
Methylene chloride	M	< 1	6.2	1.95	NC					NC
Methyl ethyl ketone	D	2	7.2	2.6	1.95	4.1				NC
Octane	M	3	3.9	12?	2.2			NC	5.82	13.50
Pentane	D	0.5	2.35	1.9	0.95			2.42	1.54	NC
Tetrachloroethylene	P	< 0.5	9.9	NC	NC	4.38				13.5

TABLE XXX (Continued)

	Certainty of identification	Approx. relative quan.	Carbowax	Apiezon 4	Triscyano 58	QF1 61	QF1 62	DEGS 60	Alcon 56b	Silicone grease 63
Tetraethylsilicate	M	< 0.3	18.7	NC						24.0
Toluene	P	1	11.3	9.42				NC	NC	10.4
Trichloroethylene	P	< 1.0	18.6		10.8					7.47
Unidentified early peaks, probably mixture of C_2, C_3, and C_4 alkanes and alkenes		20	1.44	0.6 0.95	1.00	0.81 0.98		2.42	0.76	1.0

TABLE XXXI

IDENTIFICATION OF COMPOUNDS FROM CO_2 TRAP 2ND DAY (7/19 /66

	Certainty of identification	Approx. relative quan.	Carbowax 5	Apiezon 4	Triscyano 58	QF1	DEGS 60	UCON 56a	Silicone grease 63
Acetone	D	300	5.2	1.9	13.2	2.72	8.40	4.65	1.93
Isopropanol	D	5	8.6	1.9	13.2				2.4
Ethanol	D	4	9.1	1.7	14.7		9.3	6.7	2.00
Methanol	P	4		1.22?	NC		7.6		NC
Carbon tetrachloride	P	0.3	NC	4.7	5.5			10.0	
Chloroform	M	0.3	10.0	3.22					
Cyclohexane	P	0.6	3.32		2.3		7.88		
1,2-Dimethylcyclohexane	M	< 0.1			4.7				
Ethylbenzene	M	< 0.1				7.52		18.4	
Heptane	M	4.0	NC	NC	1.5				
Hexamethylcyclotrisiloxane	M	< 0.4	4.2					6.7	13.7
Hexane	P	1.2	2.6		1.15				
2-Hexene	P	< 0.1	2.7 2.8	3.22	1.38				
Methylcyclopentane	M	< 0.1	NC	NC	NC				1.95
Methylene chloride	M	0.6	6.3	1.9	NC				2.4
Methyl ethyl ketone									
Octane	M	1	NC	NC	2.3			NC	13.7
Pentane		0.1	2.4	1.9	NC		2.3	NC	
Tetrachloroethylene	M	< 0.5	10.0				NC	NC	13.7
Toluene	P	1.0	11.5	NC	NC			13.0	10.85
Trichloroethylene	M	< 1.0	18.6		11.0				7.55?
Unidentified early peak, probably mixture of C_2, C_3 and C_4 alkanes and alkenes.		20	1.78	1.44	0.7		2.38	0.53 0.73	

TABLE XXXII

LIQUID NITROGEN TRAP 1ST DAY (7/19/66)

Compound	Certainty of identification	Approx. relative quan.	Retention times: Carbowax	Apiezon	Triscyano	QF-1 61	QF-1 62	Degs 60	Ucon 60b	Silicone Grease 63
Acetone	D	1000	5.42	1.9	12.73	2.60	2.53	8.6	3.75	2.27
Isopropyl alcohol	D	200	8.95	1.9	12.73	1.43		9.61	6.88	2.27
Ethanol	D	180	9.2	NC	14.4	NC		9.61	NC	1.95
Methanol	P	100	NC	1.2	12.73	1.23		NC		1.60
Carbon tetrachloride	P	4	5.42	NC	5.6				10.0	NC
Cyclohexane	P	1	NC	5.1	NC	1.73			3.75	
1,2-Dimethylcyclohexane	P	< 0.5	NC		4.6					31.2
Ethylbenzene	P	< 0.5	NC		2.9		7.5		18.37	13.42?
Heptane	P	3	3.0	NC	NC	2.2				8.40
Hexamethylcyclotrisiloxane	M	< 1	NC	6.6?	5.6				6.69	13.42
Hexane	D	36	2.7	3.0	1.16			3.10		
2-Hexene	M	< 0.5	2.7	1.35,1.7	NC					
Methylcyclohexane	P	< 0.5	3.85	NC	NC	2.2				
Methylcyclopentane	P	1	3.0	3.7	1.9	1.73				
Methylene chloride	P	0.6	6.4	1.9?	9.1	1.56				2.27
Methyl ethyl ketone	D	3	7.45	2.42	18.0			13.0		
Octane	P	3	3.85		NC				6.01	13.42
Pentane	P	1.2	2.2	1.9	0.90	NC		NC	NC	NC
Trichloroethane	M	< 1	5.42	3.7						
Early unidentified peak, probably mixture of C_2, C_3, and C_4 alkanes and alkenes.		9	1.95 2.2	1.2 1.44		0.90	0.81 0.98	2.18 2.53	0.86 0.94	0.70 0.98

TABLE XXXIII

LIQUID NITROGEN TRAP 2ND DAY (7/19/66)

	Certainty of identification	Approx. relative quan.	Retention times: Carbowax 5	Apiezon 4	Triscyano 58	QF-1 61	QF-1 62	DEGA 60	UCON 56	Trion X 63	Silicone grease 64
Acetone	D	125	5.47	1.92	12.8	2.60	2.75	8.6	3.75	0.90	2.55
Isopropyl alcohol	D	10	8.90	1.92	12.8	1.43		9.77	6.76		2.55
Ethanol	D	5	9.2	NC	14.7	1.56		9.77	6.70		1.90
Methanol	D	3	8.4	1.2	12.8	NC		NC		NC	1.64
Carbon tetrachloride	P	3	5.47	NC	5.6				NC		6.32
Cyclohexane	D	0.8	NC	5.0?	2.45	1.73			3.75		
1,2-Dimethylcyclohexane	M	<0.5	NC	NC	4.58						31.24?
Ethylbenzene	M	<1	NC	NC	2.89		7.52			5.14	13.90?
2-Ethyl-1-hexene	M	<1	3.68	NC	NC	2.75	2.75				
Heptane	P	3	3.0	NC	1.62	NC				0.78	8.90?
Hexamethylcyclo-trisiloxane	P	<1	NC	6.7?	5.6				6.74?		13.68?
Hexane	P	8	2.6	3.15	1.20			NC			
2-Hexene	D	1	2.6	3.15	1.72	1.56					
Methylcyclohexane	M	1	3.68	NC	2.45	NC					
Methylcyclopentane	P	1	3.0	3.7	1.8	1.73					2.55

TABLE XXXIII (CONTINUED)

	Certainty of identi-fication	Approx. relative quan.	Carbowax 5	Apiezon 4	Triscyano 58	QF-1 61	QF-1 62	DEGA 60	UCON 56	Trion X 63	Silicone grease 64
Methylene chloride	P	2	6.3	1.9?	9.1	NC					2.55
Methyl ethyl ketone	M	1.5	7.3	NC	NC						
Methyl isobutyl ketone	M	$\leq$0.5		5.9	18.6						
Octane	P	<0.5	3.68	NC	NC				6.00		13.68?
Pentane	P	1.5	2.2	1.9	0.90	NC		2.53	NC		2.55
Toluene	M	<0.5	NC	9.0?						2.9?	10.65?
Trichloroethane	M	<1.0	5.47	3.7							
Early unidentified peak, probably mixture of C_2, C_3, and C_4 alkanes and alkenes		60	1.95, 2.2	1.2, 1.44	0.58, 0.65	0.60, 0.62, 0.78, 0.88, 0.98	0.57 1.00	2.18, 2.42	0.73 0.89	0.31, 0.40	1.20, 1.23, 1.28

TABLE XXXIV

ANALYSIS OF GRAB SAMPLES

Compound	Conc. mg/ml	
	7/18/66	7/19/66
Acetone	0.004	0.005
Carbon tetrachloride	$8 \cdot 10^{-7}$	$8 \cdot 10^{-7}$
Ethanol	$2.6 \cdot 10^{-6}$	$1.6 \cdot 10^{-6}$
n-Hexane	$< 2 \cdot 10^{-8}$	$< 2 \cdot 10^{-8}$
Methylene chloride	$< 2 \cdot 10^{-6}$	$< 2 \cdot 10^{-6}$
n-Pentane	$1.4 \cdot 10^{-7}$	$< 2 \cdot 10^{-8}$
2-Propanol	$6.4 \cdot 10^{-6}$	$2.9 \cdot 10^{-6}$
Chloroform	$6.6 \cdot 10^{-6}$	$6.6 \cdot 10^{-6}$
Cyclohexane	$< 0.2 \cdot 10^{-7}$	$< 0.2 \cdot 10^{-7}$
n-Heptane	$< 0.2 \cdot 10^{-7}$	$< 0.2 \cdot 10^{-7}$
Methylcyclopentane	$< 0.2 \cdot 10^{-7}$	$< 0.2 \cdot 10^{-7}$
Methyl ethyl ketone	$< 2.0 \cdot 10^{-7}$	$< 2.0 \cdot 10^{-7}$
n-Octane	$< 1.0 \cdot 10^{-7}$	$< 1.0 \cdot 10^{-7}$
Toluene	$< 4.0 \cdot 10^{-7}$	$< 4.0 \cdot 10^{-7}$

12.0 CONCLUDING REMARKS

Ten materials used in the Langley Integrated Life Support System have been examined as possible sources of atmospheric contamination. It was found that they could contribute to atmospheric contamination principally by outgassing of solvents and plasticizers, and by decomposition of various additives such as plasticizers. The Tygon tubings, solder lug insulation, and aluminum foil tape gave off a considerable number of compounds, such as halocarbons, which could contribute to physiological distress.

Several atmospheric samples from the ILSS were obtained and analyzed by gas chromatography for contaminants. There was some correlation between the contaminants found in these samples and the compounds given off by the ten materials that were tested.

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